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EDGEWOOD ARSENAL CONTRACTOR REPORT

EM-CR-74052

(EA - 4D11)

FORMULATION OF HAZARD EVALUATION
INDICES FOR PYROTECHNIC PROCESSES

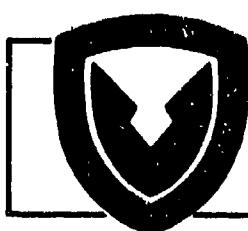
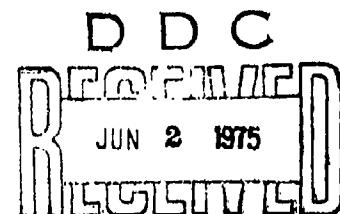
by

W. R. Nestle

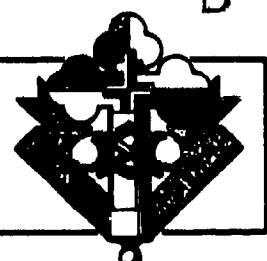
March 1975

NASA NATIONAL SPACE TECHNOLOGY LABORATORIES
General Electric Company
Engineering and Science Services Laboratory
Bay Saint Louis, Mississippi 39520

Contract No. NAS8-27750



DEPARTMENT OF THE ARMY
Headquarters, Edgewood Arsenal
Aberdeen Proving Ground, Maryland 21010



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PREFACE

The work described in this report was authorized under MIPR B4030. It was performed at the NASA National Space Technology Laboratories (NSTL) for the Edgewood Arsenal Resident Laboratory (EARL) and NASA-NSTL by the General Electric Company under Contract No. NAS8-27750. This task was initiated 25 September 1973 and completed 20 September 1974.

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FORMULATION OF HAZARD EVALUATION INDICES FOR PYROTECHNIC PROCESSES

1.0 INTRODUCTION

1.1 Objective. The objectives of this program were to design test apparatus and develop laboratory test procedures for evaluating the hazards associated with the pneumatic transfer and mixing of pyrotechnic materials. Specifically, the apparatus were developed to determine the velocity threshold for ignition, friction sensitivity, and energy release characteristics under nominal processing conditions. A secondary objective was to determine these characteristics for the materials contained in two representative colored-smoke mixes:

- Violet Smoke IV, Drawing No. B143-5-1
- Green Smoke IV, Drawing No. B143-2-1

1.2 Authority. The authority for this project is TWR EA-4D11 issued 26 September 1973 and defined by the project support plan for Hazards Evaluation Processing Indices dated 12 December 1973.

1.3 Background. A survey of available literature indicates that, although considerable research has been done in the field of pneumatic conveying of solids, test apparatus for determining associated hazards and written theory on the subject are still in the developmental stage. The engineering required for the design of a pneumatic conveying system is extensive. For example, materials can be conveyed pneumatically with a broad range of fluid velocities. The required velocity to maintain the material suspended in the stream is a function of particle size, density, shape, and other physical characteristics such as the length of the system and whether the direction of flow is horizontal or vertical.

Materials conveyed in pipes are continuously subjected to potential ignition or initiation stimuli, particularly as a result of friction and impact. Materials whose physical characteristics require high air velocity for transport necessarily possess higher energy at impact. In making a determination of the suitability of a material for processing in a pneumatic conveying system, it is of primary importance to determine the impingement velocity threshold of ignition or to determine whether ignition will occur within a nominal range above the minimum required transport velocity. To evaluate these characteristics, it was necessary to develop apparatus to determine the velocity threshold of ignition and friction sensitivity and to develop sensitivity tests for the materials which would allow these characteristics to be expressed in terms of force, energy, and velocity.

The mass of suspended particles within a pneumatic conveying system are also highly susceptible to ignition from side wall impact, sliding friction, or from the discharge of static electricity resulting from particle movement in the system. Should ignition occur, the sudden increase in pressure and temperature can cause system rupture at that point or effect propagation to other parts of the system. A determination of magnitude of energy released and the kinetics involved is necessary for development of suppressive and relief

devices for system protection. The sensitivity to ignition of pyrotechnic smoke mixes has been studied on a laboratory scale with such devices as the Hartmann Chamber for determining minimum concentration and energy for ignition and the Parr bomb calorimeter for determining heat of combustion. A need exists for a test apparatus to determine the energy release characteristics of pyrotechnic materials in which a sample size representative of a factory processing environment can be duplicated. Such a device would permit comparison of large scale tests with theoretical and laboratory test data. The intent of this project is to extend the scope of hazard evaluation procedures to the study of material initiation and energy release characteristics under simulated pneumatic processing conditions.

2.0 TECHNICAL APPROACH

2.1 Impingement Studies. It was determined during the study phase of this project that pneumatic transport systems were operated at air/material velocities of 100 to 150 feet per second. This range was determined to be satisfactory for maintaining stream integrity for the materials considered in this project. To provide a reasonably safe criterion for pneumatic transport, it was decided that a material exhibiting no impingement reaction at a velocity of twice the conveying velocity (providing an energy safety factor of at least four) could be considered safe for pneumatic transport. In addition to the possibility of impact and friction induced initiation, moving materials build up electrostatic charges due to triboelectrification among the materials or on impact with the container; a spark caused by such effects could ignite the dust/air mixture causing a fire or explosion. It is necessary to determine the effect of the material velocity on the static charge buildup. The impingement apparatus based on the above criteria was designed to propel particles at velocities of 50 - 300 feet per second, to provide a means of testing the effect of impingement of various materials at variable angles of impact, and to measure the gross static charge buildup in the fluidized system.

The apparatus and peripheral equipment are shown pictorially and schematically in figures A-1 to A-11. The apparatus is composed of four major assemblies; the pneumatic system, sample storage and injection system, target holder and dust collector, and the instrumentation and control system. The subassemblies and components of these assemblies are described in the following paragraphs.

2.1.1 Pneumatic System. The pneumatic system components are contained in two instrumentation racks with a portable valve control unit to provide for either remote or local operation of the system, depending on the hazard involved, see figures A-2 to A-5.

- Cabinet "A" contains the following:
 - A regulator to reduce the base air pressure and regulate air to the accumulator.
 - A hand vent valve to reduce pressure.
 - A gage to aid in setting tank pressure.
- Cabinet "B" contains the following:
 - A hand shut-off valve to the accumulator to isolate the tank from the regulator after final adjustment.

NOTE: Figures A-1 through A-22 are to be found in appendix A.

- A hand vent valve to reduce tank pressure.
- A gage to read accumulator pressure.
- A full-port solenoid valve down stream of the accumulator to release air and drive the dust sample down the ejector tube.
- A second solenoid connected in parallel with the first to vent air through a gas ionizer device and then through the ejector tube. Air was vented through this system after each test to neutralize the static charge buildup in the ejector tube and free the dust which clung to the tube side walls. The gas ionizing device used was a 3M Company Model 906 ionizing air nozzle containing 20 millicuries of Polonium-210.

2.1.2 Sample Storage and Injection System. A rotating sample storage table contains 16 Teflon storage wells and 16 Teflon plugs. Each storage well has a capacity of 400 mg of material (see figure A-6).

A one RPM 50 in-lb torque motor with chain and sprocket drive was used to turn the table. It was connected in series with a momentary start switch and a microswitch cutoff. Once the motor was activated by the momentary switch the table would rotate dropping a sample into the venturi block chamber and would continue to rotate until a plug closed the venturi block opening. This system of sample injection was later modified. (See discussion of equipment development problems, paragraph 3.1.1.)

The ejector tube was a 1/4-inch O.D., 0.125-inch I. D., Plexiglas tube 3 feet long. The tube was welded to the venturi block, extended into the collector box and terminated 1/2-inch from the target face. Attached to the ejector tube is a fixture for holding two optical transducers and associated light sources. The transducers were set exactly 1 foot apart with the optical beam perpendicular to the longitudinal axis of the tube. The optical transducers were used to measure the velocity of the dust cloud, by determining the time of travel between the sensors.

2.1.3 Target Holder and Dust Collector. The dust collection box is 3.8 cubic feet in volume, with a 150 square inch dust collecting vent screen and a bottom clean out hatch. The target holder and removable anvil are mounted inside the collector on a movable arm which allows the target to be set at angles from 0 to 45 degrees to the direction of particle flight. The target anvils were removable so that the type of material and surface roughness could be varied.

2.1.4 Instrumentation and System Control. The instrumentation and control equipment are shown in figures A-2 to A-10. Differential voltage time curves from the optical transducers were recorded by a Polaroid camera attached to the dual beam oscilloscope. (See figure 1 for typical curve.)

2.1.5 Testing Rationale/Procedure. The test equipment used in the conduct of impingement testing is shown in table 1.

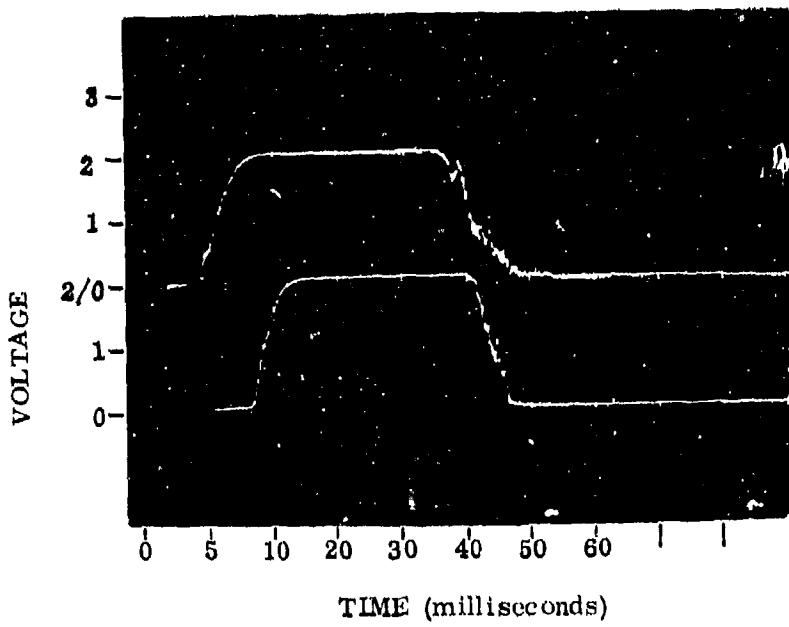


Figure 1. Typical Voltage Rise Curve for Photo Transducer

Table 1. Test Equipment Used in Impingement Testing

| Item | Manufacturer | Model |
|------------------------|------------------|---------|
| Electronic Counter | Hewlett Packard | 5245L |
| Power Supply | Sorenson | QB6-8 |
| Power Supply | Power Design | TW-4005 |
| Dual Beam Oscilloscope | Tektronix, Inc. | 555 |
| Motion Picture Camera | Fastex | WF-4 |
| Camera | Polaroid | C-12 |
| Camera | Polaroid | 100 |
| Camera | Linhof-Technika | 5 |
| Motion Picture Cameras | Mitchell Monitor | 500 |

The circuits for camera and valve operations are shown in schematic representation in figures A-7 through A-9. The control circuits were arranged through a series of time delay relays which activated the camera and the main gas valve, synchronizing the arrival of the dust at the target and the attainment of desired frame speed. The time delay relays were adjustable to allow for a broad range of particle velocities and frame speeds.

Measurement of particle velocity was made using the optical transducers to trigger and stop an electronic counter. The photometer output voltage is a logarithmic function of the incident light intensity, and the incident light intensity is an inverse exponential function of the density of the particle cloud in the beam. The overall response is thus approximately a linear function of the particle concentration. Both sensors were set to trigger the counter at the same voltage level under assumption that the concentration of the cloud remains essentially constant over the one foot distance separating the sensors. Polaroid photographs of the optical sensor voltage versus time were also made as a check on counter time; see figure 1. This system did not perform as expected, probably due to turbulence and other physical properties of the materials. Problems with velocity measurements are detailed in paragraph 3.1.1. Figure A-10 shows the Fastex Camera aligned for particle cloud photography.

2.1.6 Reaction Criteria. A reaction initiation was considered to have occurred if a flash or sparkle occurred and/or a small portion of the material was consumed.

2.1.7 Sample Preparation. The following procedure was used for all tests performed on this project:

- All sample materials were dried at 75°C in a Blue-M, Model IR100, Friction Aire oven for a minimum of 22 hours to assure uniform moisture content.
- After oven drying, sample materials were kept in a desiccator to assure a dry condition.
- The laboratory was equipped with an air lock to minimize temperature and humidity changes. It was air conditioned and dehumidified to minimize moisture absorption by the material during handling.
- All samples were sieved through a No. 200 (US Standard Series) screen prior to drying and testing to minimize particle size variations.
- All samples were weighed to within \pm 0.5mg on a precision analytical balance.

2.1.8 Impingement Test Procedures. The test was conducted as follows:

- The test equipment setup is shown in figure A-1.
- The selected quantity of sample material was weighed and placed in each of the 16 sample storage wells. It was found during the experiment that 100 mg samples provided the most consistent results.

- The rotating table motor drive was activated and a sample was deposited in the venturi block.
- The tank pressure was set to the selected value and the tank was isolated from the regulator by closing the inlet valve.
- Laboratory lights were switched off to provide maximum contrast for observation.
- The main valve was opened for three seconds providing the force necessary to drive the material down the ejector tube against the target.
- Observations for evidence of ignition were recorded on the test data sheet.
- The static charge present and the transit time of travel of the dust cloud between the optical transducers was recorded. For each series of 10 tests, five each determinations of the static charge along the ejector tube and at the target were recorded.
- The deionizing valve was actuated and any residual dust was removed from the ejector tube. The static charge level was reduced by this operation to an acceptable level.

2.2 Friction Sensitivity Apparatus. To determine the friction initiation characteristics of materials it was considered important to construct an apparatus with a controllable striking force and with the capability for measuring the friction initiation pressures as a function of the sliding velocity. The apparatus and peripheral equipment designed for this purpose are shown pictorially and schematically in figures A-12 to A-17. The apparatus consists of a variable weight pendulum mounted in a pyramidal framework. A solenoid mechanism capable of securing and releasing the pendulum from any height within the 180° limit of travel is provided. By varying the height, the drop velocity can be varied from 0 to 28 feet per second at impact. The pendulum weight is variable from 1 to 25 pounds in one-half pound increments to provide a variable impact force.

The test samples were positioned between the flat end of a 1/2 inch diameter right circular cylinder and a sliding bar. In operation friction is created by the relative motion between the sliding bar and the right circular cylinder. The cylinder is held in a fixture above the bar with a load measuring cell clamped between it and a threaded rod used to apply a known load to the sample. The sliding bar extends from the rear of the base into the path of the pendulum. A linear velocity transducer is attached beneath the bar to measure the velocity when struck by the pendulum. The cylinder is clamped with its axis of symmetry perpendicular to the broadest plane of the sliding bar with test material between the two surfaces. The amount of force holding the two members in contact is monitored by a resistance strain gage type load cell, Sensotec Model LCFB-20 with a load capacity of 0 to 500 pounds. The force is made variable by the amount of torque applied to the load screw installed axially above the cylinder. The sliding bar is restrained from movement in all but one direction, that of the pendulum swing, by a drawer type member referred to as a specimen tray. The aft edge of the tray is struck by the head of the pendulum after being released from a known angle as indicated by the degree wheel. The degree wheel is slotted

circumferentially and marked in degrees from zero to 180. The pendulum arm and release mechanism is a solenoid operated draw pin mounted on a plate which is attached to the degree wheel with a locking bolt. The fixture can be adjusted to hold and drop the pendulum from a specified height. The pendulum transfers energy to the sliding bar on impact. The velocity attained by the sliding bar from the pendulum is monitored by a Trans-Tex Model 114 velocity transducer which consists of a moving magnet set in a stationary coil. The magnet is attached to the tray and sliding bar arrangement such that tray motion produces a voltage output proportional to the velocity.

2.3 Closed Vessel (Modified Parr Bomb) Apparatus. During a previous project a closed vessel with a working pressure of 75 psi was used for large scale calorimetric tests on explosive materials.¹ The test data showed good correlation with theoretical calculations. The vessel, sample holder and method of initiating combustion were modified and used for testing pyrotechnic materials on this program. The apparatus and peripheral equipment designed for these tests are shown in figures A-18 to A-22. The vessel is 4 feet in diameter with a volume of 37.66 cubic feet. The device shown in figure A-20 was used to hold and ignite the material samples. The center core of the holder is a 3/4-inch diameter cardboard tube with four holes 90° apart every 1/2-inch. The tube was wrapped with six coils of .012 inch stainless steel wire which was connected to the hot wire blasting machine shown in figures A-21 and A-22. The test material was placed around the center core. The top of the container was closed and the hollow center core was open to allow burning gases to escape. Chromel/alumel thermocouples were located to measure the tank air temperature, the sample holder temperature and the temperature of the escaping gases. Internal tank pressure was measured by an MB Electronics Model 151-ESC-194 pressure transducer. The output of all instruments was recorded on a Gould Brush Model 260 recorder. Gas samples were taken when the tank pressure reached its maximum levels.

2.3.1 Sample Preparation and Testing. Sample drying, sieving and general handling methods used for these tests are the same as noted in paragraph 2.1.7. Upon completion of the sample preparation phase the bulk material was weighed out and loaded into the sample holder shown in figure A-20. The material weights for each test are shown in table 4; the sample size was based on anticipated pressure as calculated from an equation developed by William S. Filler² and modified by F. S. Schultz:¹

¹F. S. Schultz, Static Pressures Investigation for the Chemical Agent Demilitarization System EA-FR-2B01, June 30, 1973.

²William S. Filler, Post Detonation Pressure and Thermal Studies of Solid High Explosives in a Closed Chamber. Sixth Symposium on Combustion 1956, pgs. 648-657.

The equation is as follows:

$$P = \frac{H(\gamma-1)}{V}$$

where

P = pressure rise

H = heat added to the gas

γ = ratio of specific heats of air (C_p/C_v)

V = volume of container

For the low pressures and temperatures expected, γ is approximately 1.4. When P is expressed in psi, H in kilocalories/gram, and V in cubic feet the equation could be expressed as follows:

$$P = \frac{3844 W H}{V}$$

where

P = pressure (pounds/square inch)

W = weight of explosive (pounds)

H = heat of combustion (kilocalories/gram)

V = volume (cubic feet)

The loaded sample holder was located in the approximate center of the vessel. The tank was sealed and the sample ignition wires connected. Ignition power was applied for three seconds and in all tests the igniter wires burned through in this period of time. When the tank reached peak pressure a sample of the combustion gases was taken. Pressure and temperatures were monitored for thirty minutes after ignition. The tank was then purged with air and the condition of the tank and sample examined.

3.0 RESULTS

3.1 Particle Impingement Studies. Test results for each material are summarized in tables 2, 3 and 4.

3.1.1 Equipment Problems. A number of problems were encountered in the development phase of this project; this section will note and discuss the most important. The greatest difficulty encountered was in achieving the desired particle cloud velocities and measurement of this parameter. Problems encountered with the optical sensors used to measure transport times across a known distance and the mechanical aspects of the apparatus which affected the dust cloud velocities are:

Table 2. Impingement Data Violet Smoke IV B143-5-1

| Nominal Velocity Ft/Sec | Average Velocity Ft/Sec | Average Electrostatic Charge 10^{-10} Coulomb | | | Target Material | Observed Reaction |
|-------------------------|-------------------------|---|-------|-----------------|-----------------|-------------------|
| | | Target | Tube | Target \angle | | |
| 200 | 206.8 | +22 | -.01 | 0° | Steel | None |
| | 205.6 | .05 | -.025 | 45° | Steel | None |
| | 253.1 | .4 | -.021 | 0° | Steel | None |
| | 249.1 | .15 | -.026 | 45° | Steel | None |
| | 306.2 | .21 | -.044 | 0° | Steel | None |
| | 302.4 | .1 | -.034 | 45° | Steel | None |

Table 3. Impingement Data, Green Smoke IV B143-2-1

| Nominal Velocity Ft/Sec | Average Velocity Ft/Sec | Electrostatic Charge 10^{-10} Coulomb | | | Target Material | Observed Reaction |
|-------------------------|-------------------------|---|--------|-----------------|-----------------|-------------------|
| | | Target | Tube | Target \angle | | |
| 200 | 208.5 | .16 | -.02 | 0° | Steel | None |
| | 211.6 | .17 | -.01 | 45° | Steel | None |
| | 250.3 | .17 | -.009 | 0° | Steel | None |
| | 251.1 | .16 | -.0084 | 45° | Steel | None |
| | 308.4 | .26 | -.01 | 0° | Steel | None |
| | 305.6 | .26 | -.007 | 45° | Steel | None |

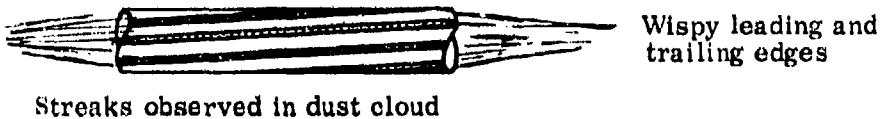
- Location of the Optical Sensor Circuit Amplifier Elements - In the original design the optical sensor leads ran from the mounting blocks to a panel in instrumentation Cabinet "A". During checkout tests it was noted that operation of the main air solenoid frequently triggered the electronic counter. Shielding of the sensor leads did not alleviate the problem. It was necessary to mount the amplifier elements directly under the optical elements in the shielded box.
- Light Tube Effect - The Plexiglas mounting blocks and ejector pipe acted as a light tube when the laboratory lights were on during a test. Sufficient light was present from this external source that the sensors could not distinguish a change in opacity during the dust transfer, and the electronic counter failed to trigger.

Table 4. Impingement Data Smoke Components

| Material | Nominal Velocity Ft./Sec | Average Velocity Ft./Sec | Electrostatic Charge 10^{-10} Coulomb Target Tube | Target Angle | Target Material | Observed Reaction |
|--------------------|--------------------------|----------------------------------|---|--|--------------------------|--|
| Sulfur Powder | 300 | 308.8 | .08 .05 | 45° | Steel | None |
| Aluminum Powder | 300 | 304.6 300.9 | +8.4x10 ⁻⁸ +1.0 | -.01 -0.6 | 45° 45° | Steel Aluminum |
| Potassium Chlorate | 300 | 289.0 290.1 | -.6 -.46 | Charge was erratic plus to minus during test | 45° | Steel Aluminum |
| Benzanthrone | 300 | 290.3 299.4 | Erratic -.5 | +.13 .2 | 45° 45° | Steel Aluminum |
| Sodium Bicarbonate | 300 200 | 303.5 299.5 195.3 219.4 | .29 .08 .65 .11 | -.03 -.04 Not taken Not taken | 45° 45° 45° 45° | Steel Aluminum Steel Aluminum |

The problem was solved by use of a cardboard tube over the sensors and black paint on the tube ends to block the entrance of external light.

- Fine Powder and Dye Materials - Fine grained powdery materials, particularly sulfur and benzanthrone (and to a lesser extent dye materials), of the smoke compositions adversely affected the optical sensor voltage levels. These materials clung tenaciously to the tube side walls during transit. This increased the opacity of the tube and resulted in sensor voltage levels higher than the calibrated base. Conduct of subsequent tests prior to flushing the tube would cause counter triggering at reduced concentration levels and produced erroneous transport time measurements.
- Cloud Density - Analysis of high speed motion pictures of the moving particle cloud revealed that the cloud was often composed of variable concentration streaks as shown in the following sketch.



The use of optical sensors to measure the cloud velocity requires that the cloud be of uniform concentration or opaqueness and that the sensor respond to the leading edge of the cloud. In practice, neither of these conditions are met. The relative position in the cloud of a concentration sufficient to trigger the first sensor changes during transit to the stop sensor, thus accounting primarily for velocity differences observed from test to test. The diffused leading cloud edge also contributes to system inaccuracy.

- Dual Trace Oscilloscope - A dual trace oscilloscope was used to provide a secondary measurement of the transit time and to provide a picture of the voltage rise curve from the optical sensors. A typical curve is shown in figure 1. In all tests the transit time measured by use of the oscilloscope was 10 to 20 percent less than the electronic counter time. The electronic counter time was recorded and used to determine the cloud velocity.
- Sample Storage and Injection System - In the original design the Teflon storage blocks were 3/4 inch long with a cavity tapering from 1/4 inch diameter at the top to 1/8 inch at the exit end. During transit of the storage block across the venturi opening, the sample, instead of dropping under the force of gravity into the venturi opening, would cling to the sides of the sample storage block. A modification to provide a right circular cavity 1/4 inch in diameter for the sample block worked well with granular materials but was unsatisfactory for soft powdery materials. Since it was not possible to further enlarge the cavity a redesign of the air injection system was effected so that an air stream forced the sample into the ejector tube. The material adhesion problem was due both to electrostatic attraction between the sample and the Teflon sample holder and to the cohesive force of the material.

- Sample Holder Orifice - In the original design the air stream entered the venturi block from the back in line with the direction of flow down the ejector tube. The change to the system necessitated by the problem of sample injection required routing the air through the sample storage blocks. Slight differences in sample block orifices caused significant variations in air velocity. Comparative flow tests through the sample holders were made in an attempt to identify those yielding consistent velocity readings. No two holders were sufficiently alike, requiring abandonment of the multiple sample turntable concept. The air injection line was modified to allow placement of the sample directly in the line. A time study revealed that this method of sample injection resulted in only a 10 percent loss in testing time. The problem would have been more severe if the testing were of necessity done remotely. This problem could be reduced if the sample holders were metallic and made to precise tolerances.
- Dust Filtering System - The air/dust mixture which entered the dust collector box was vented through ten layers of cheese cloth to remove any particles from the air as it was exhausted into the laboratory atmosphere. This proved to be inadequate to remove the finely divided materials. Further use of this system should incorporate a powered air exhaust line near the vent to remove the dust from the laboratory.
- Flow Duration - The air stream was allowed to flow for approximately three seconds to assure that there was a constant dynamic force behind the sample during the entire transit period. The sample, however, was in the ejector line only a matter of milliseconds. This created innumerable problems in achieving dynamic stability of any of the instrumented parameters. To achieve stable flow, it is recommended that samples of sufficient size be used in tests with powdered materials.
- High Speed Motion Pictures - One of the primary reasons for constructing the impingement apparatus of Plexiglas was to provide a visual means for the study of the particle flow and impact phenomena. High speed motion pictures were obtained to provide a secondary means of measuring particle velocity. Using both the Mitchell and Fastex cameras, a series of color film strips were made of the particle cloud between the exit end of the tube and the target. The pictures were taken against a ruled background to provide a measurement of distance. It had been anticipated that some distinctive shape or particle could be identified and tracked through several frames to establish a particle velocity. Tests were run using both powder and granular materials with some test samples containing particles of contrasting color for identification. Examination of the film frame by frame and at normal speed showed the particle cloud leading edge to be a light, scattered wisp of smoke gradually becoming more dense as the main body of powder passed, with the trailing edge less well defined. Film strips were taken at 500 to 4000 frames per second. In none of the film examined was it possible to distinguish individual particles or formations from one frame to the next which could provide velocity or flow measurements. Film strips showed the main body of the cloud to be composed of long streaks of varying density. The effect of this configuration on the velocity measurement is an previously discussed under the heading of cloud density.

- Still Photography - Attempts were made to record photographically the light flashes occurring at impact. Pictures were made with both the Polaroid and Linhof-Technika cameras. None of the film used was sufficiently sensitive to record the pinpoint of light. Other investigators have recorded similar flashes using, ASA 10,000 film, which is no longer available, and further attempts to capture the phenomenon on film were abandoned.

3.1.2 Observations. A summary of the test results and a list of the materials tested are shown in paragraph 3.1 and tables 2 to 4. Each summarized data point is the average of 10 tests. Impingement tests were conducted with two smoke compositions, Violet IV and Green IV, at velocities of 200, 250, and 300 feet per second against a steel target. Ten tests at each velocity were run against the target set at 0 and 45 degrees with respect to the direction of flight. Impingement tests against flat and angled targets were conducted to observe the difference, if any, in the initiation characteristics of particles from direct impact and sliding impact.

Impingement tests were conducted with smoke components at velocity of 300 feet per second, against both steel and aluminum targets set at 45 degrees to the direction of impact. Electrostatic measurements were made at the longitudinal center of the ejector tube and at the target face. In each series of 10 impingement tests five measurements were made at each point. The electrostatic charge values shown in the summary sheets are the average of five tests.

None of the materials tested exhibited an explosive or burning reaction due to impingement on steel at transport velocities up to 300 feet per second. Although this would indicate a margin of safety twice the normal transport velocity of 100 to 150 feet per second, it should be noted that environmental conditions, material size, chemical and abrasive contaminants were under laboratory control at all times. Sodium baroconate did exhibit an apparent electrostatic spark discharge when impinged on both steel and aluminum target materials at transport velocities above 200 feet per second. A similar spark was noted during the equipment checkout phase with M-80 firecracker mix. Neither of these materials exhibited any tendency to initiate a burning or explosive reaction as a result of the static discharge. It should be noted, however, that the test samples were small and the dust atmosphere in the dust collector was less dense compared to the conditions expected in the processing mix tank or storage vessel.

Electrostatic charge measurements taken on the target during impingement showed no significant difference between those materials that did and did not exhibit a spark. A review of the electrostatic measurements reveals no significant trends among the various materials except that measurements on the target during impingement with aluminum dust were three orders of magnitude higher than the other materials tested. Since the aluminum did not exhibit a visible electrostatic spark discharge, two effects are postulated; either the metallic powder permitted increased efficiency of charge transfer to the plate, or the occurrence of spark discharge is hampered by the conductive particles. Both possibilities warrant further investigation.

3.2 Friction Sensitivity Studies. Due to the failure of a major component of the friction apparatus during checkout, no significant data was obtained.

3.2.1 Equipment Problems. During the equipment checkout phase the interchangeable portion of the sliding bar was ejected from the apparatus when the ratio of preload pressure to pendulum energy was low. In addition, the sliding bar itself overreached the 4-inch maximum travel of the velocity transducer. Two modifications were required; the interchangeable portion of the sliding bar was bolted to the main portion, and a bumper plate was added to the support block to prevent overtravel.

M-80 firecracker powder was used as the test material during checkout of the apparatus since this material was known to be friction sensitive. The material failed to react over a broad range of energy input values. Investigation of the preload record showed the preload pressure dropped to zero at impact. It was further observed that the sample material sheared at impact, part of it moving from beneath the preload cylinder onto the sliding bar. This reduction in the height of the material caused a complete loss of preload pressure with no friction force being applied to the sample during the four inch travel. The apparatus was modified by adding a plate between the preload cylinder and the sample so that the sample was distributed between two plates 4 inches long and 2 inches wide with the preload applied to the center of the upper plate. Tests made after this modification with M-80 firecracker powder mixed with a small amount of sandpaper grit produced explosive reactions at greater than 20 foot pounds of pendulum energy. During this initial period of testing the preload cell transducer failed due to an apparent internal short. A replacement transducer was not available, so testing was of necessity suspended.

3.3 Modified Parr Bomb Studies. The test results are shown in tables 5 through 8. Table 5 lists the materials tested, the size of the samples, the calculated and measured vessel pressure, the calculated and measured heat of combustion plus pertinent observations. Table 6 shows the gas analysis of the samples which burned. Tables 7 and 8 list the data extracted from the test records. Figures 2 and 3 show graphically the superimposed plots of data from the test records.

3.3.1 Observations. The original premise and technical approach to the calorimetric studies assumed that the test data would be similar in magnitude to that experienced with explosive materials which exhibit adiabatic thermochemical effects. The instrumentation was located and calibrated to record the results of complete rapid burning of the samples. The maximum pressure prediction was based on heat of combustion data derived from laboratory bomb calorimeter tests in which materials are completely oxidized. The original instrumentation included only vessel air temperature and pressure. The pressure gage was mounted 10 feet from the tank to dampen shock ringing that had been experienced in explosive tests. Observation of the vessel interior, sample residue and sample holder after the first test showed that the dye material was not oxidized in this test as had been the case in the laboratory calorimeter, and much of the energy released by the reaction was absorbed by the sample holder and hang-down rod. Analysis of the vessel pressure versus time curve indicated an apparent slow burning time which would allow some loss of heat energy to the vessel itself. No internal air temperature rise was detected.

In order to provide additional data on the energy release phenomena a thermocouple was attached to the sample holder. To determine the burning time a thermocouple was mounted to monitor the burning gases being emitted. The pressure gage was remounted directly to the tank and recalibrated to the lower expected pressure of 2 to 5 psi.

Table 5. Modified Parr Bomb Test Results

| Material Description | Sample Wt. Grams | Calculated * Pressure (PSI) | Max. Measured Static Pressure (PSI) | Pressure Rise Time Seconds | Max. Air Temp. Above Sample Holder (°F) | Alt. Temp. Rise Time (Seconds) | Experimental Thermal Energy K/CaI. | Laboratory Parr Bomb Thermal Energy K/CaI. | Laboratory Parr Bomb Heat of Combustion CaI/Br | Observations |
|-----------------------------|------------------|-----------------------------|-------------------------------------|----------------------------|---|--------------------------------|------------------------------------|--|--|--|
| Violet Smoke IV B143-5-1 | 50 | 33.7 | 2.2 | 25 | 450 | 25 | 16.5 | 149.7 | 2984 | Sample except for dye component burned |
| Green Smoke IV B143-2-1 | 50 | 27.9 | 1.5 | 60 | 430 | 60 | 15.99 | 124.4 | 2487 | Sample except for dye component burned |
| Aluminum Powder | 20 | 33.3 | 0 | 0 | 0 | 0 | 0 | 147.8 | 7389 | Sample did not burn |
| Sulfur Powder | 50 | 25.2 | 0 | 0 | 0 | 0 | 0 | 112.0 | 2240 | Sample did not burn |
| Potassium Chlorate | 40 | ** | 0 | 0 | 0 | 0 | 0 | ** | ** | Sample did not burn |
| Benzanthrone | 20 | 38.3 | 0 | 0 | 0 | 0 | 0 | 171.1 | 8556 | Sample did not burn |

* Calculated pressures were based on Paragraph 2.3.1.

** No reaction in Parr Bomb.

Table 6. Modified Parr Bomb Tests - Gas Samples Analysis

| Material | % of Sample Found | | | | | | |
|-----------------|-------------------|----------------|-----------------|----------|-----------------|-----------------|-------|
| | O ₂ | N ₂ | CO ₂ | CO | SO ₂ | NO ₂ | Dye |
| Violet Smoke IV | 19% | 77.0% | 3.2% | None | None | None | Trace |
| B-143-5-1 | | | | Detected | Detected | Detected | |
| Green Smoke IV | 19% | 76.0% | 3.8% | None | None | None | Trace |
| B-143-2-1 | | | | Detected | Detected | Detected | |

Table 7. Pressure and Temperature Profile of Green Smoke IV B143-2-1

| Time (Sec) | Tank Pressure (PSI) | Sample Holder Temperature (°F) | Air Temperature Above Holder (°F) | Air Temperature Near Wall (°F) |
|---------------|------------------------|--------------------------------------|---|--------------------------------------|
| 0 | 0.0 | 75 | 77 | 74 |
| 5 | .11 | 78 | 212 | 74 |
| 10 | .64 | 96 | 406 | 74 |
| 15 | .80 | 112 | 453 | 74 |
| 20 | .90 | 120 | 464 | 74 |
| 25 | .95 | 128 | 460 | 74 |
| 30 | .98 | 134 | 460 | 74 |
| 35 | 1.02 | 140 | 464 | 75 |
| 40 | 1.16 | 149 | 474 | 75 |
| 50 | 1.43 | 165 | 460 | 75 |
| 60 | 1.50 | 184 | 430 | 76 |
| 70 | 1.40 | 197 | 413 | 76 |
| 80 | 1.35 | 205 | 408 | 76 |
| 90 | 1.27 | 211 | 384 | 76 |
| 120 | 1.20 | 216 | 337 | 75 |
| 150 | 1.16 | 216 | 298 | 75 |
| 180 | 1.12 | 214 | 257 | 75 |
| 240 | 1.10 | 211 | 223 | 75 |
| 300 | 1.08 | 206 | 207 | 75 |
| 600 | 1.01 | 185 | 179 | 74.5 |

Table 8. Pressure and Temperature Profile of Violet Smoke IV B143-5-1

| Time (Sec) | Tank Pressure (PSI) | Sample Holder Temperature (°F) | Air Temperature Above Holder (°F) | Air Temperature Near Wall (°F) |
|---------------|------------------------|--------------------------------------|---|--------------------------------------|
| 0 | 0.05 | 70 | 67 | Data Unrecoverable |
| 5 | 0.10 | 70 | 141 | |
| 10 | 0.47 | 75 | 190 | |
| 15 | 1.48* | 102 | 410 | |
| 20 | 2.01 | 123 | 486 | |
| 25 | 2.23 | 141 | 510 | |
| 30 | 1.95 | 150 | 500 | |
| 35 | 1.80 | 159 | 480 | |
| 40 | 1.63 | 163 | 465 | |
| 50 | 1.48 | 166 | 410 | |
| 60 | 1.43 | 170 | 362 | |
| 70 | 1.38 | 170 | 327 | |
| 80 | 1.33 | 170 | 303 | |
| 90 | 1.30 | 170 | 285 | |
| 120 | 1.27 | 170 | 220 | |
| 150 | 1.21 | 170 | 179 | |
| 180 | 1.19 | 170 | 157 | |
| 240 | 1.17 | 166 | 135 | |
| 300 | 1.15 | 163 | 123 | |
| 600 | 1.05 | 149 | 112 | Data Unrecoverable |

* Slight plateau effect to pressure at 15 sec.

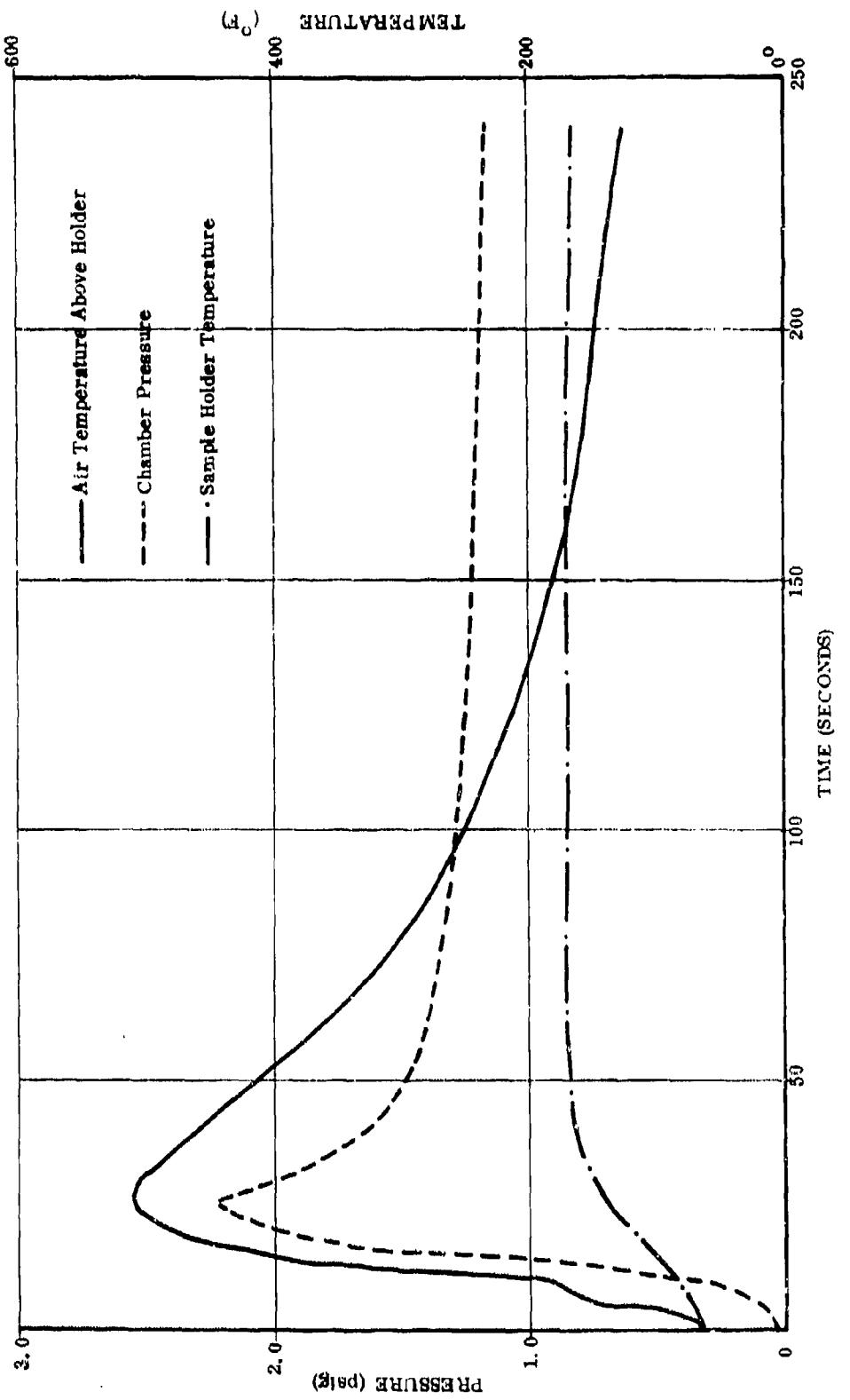


Figure 2. Modified Parr Bomb Test. Pressure and Temperature Data Violet Smoke IV B143-5-1

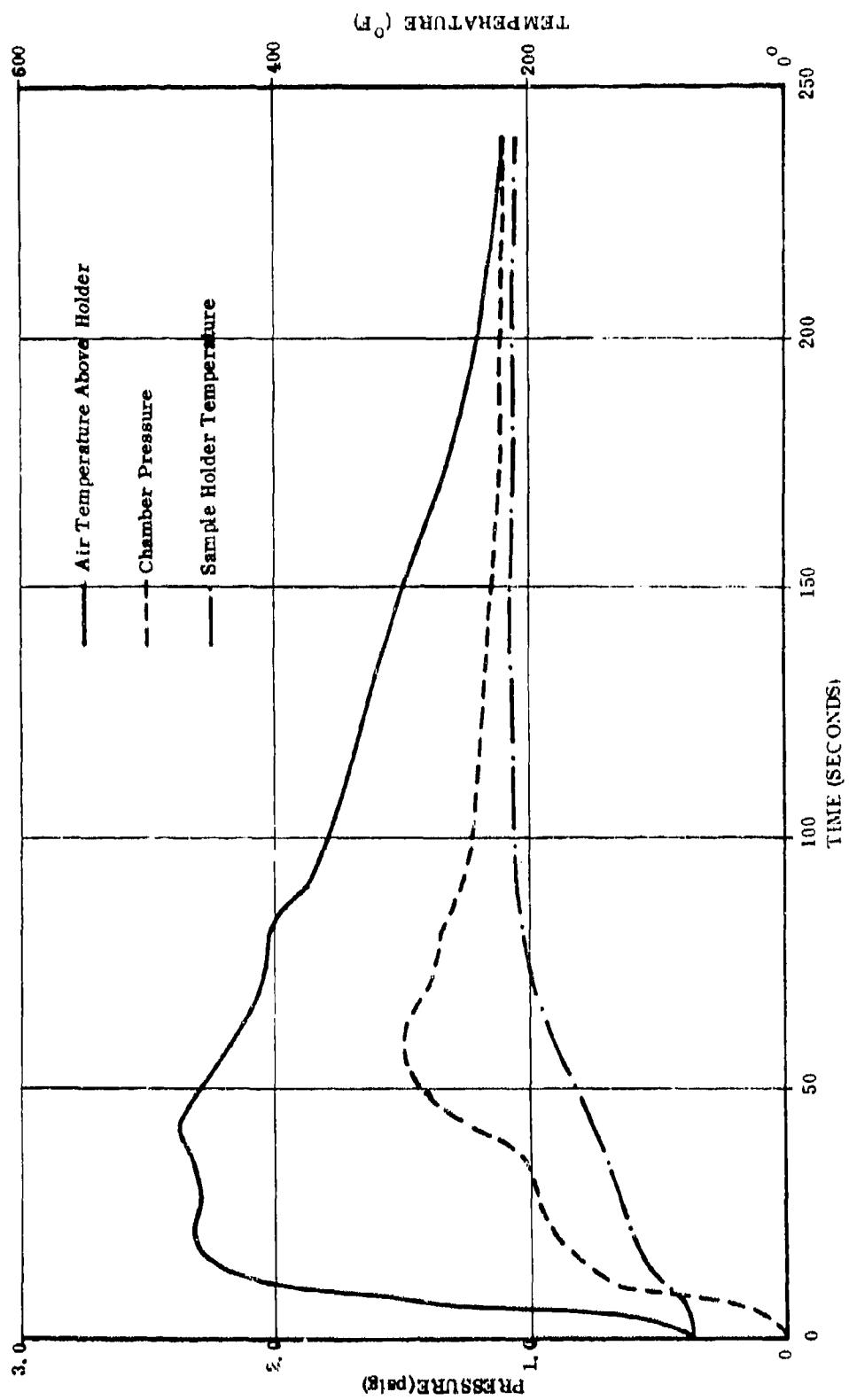
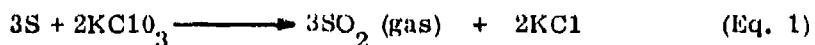


Figure 3. Modified Parr Bomb Test. Pressure and Temperature Data Green Smoke IV B143-2-1

In analyzing the data received, the following theoretical considerations and assumptions were made. The chemically active ingredients in both the green and violet smokes are sulfur (S) and potassium chlorate (KC1O₃), representing approximately 35 percent of the total mixture by weight. The smoke mixes also contain about 25 percent sodium bicarbonate (NaHCO₃) which acts as a retardant to burning and gas dispersing agent while the remaining mixture consists of colored dyes. The chemical reaction taking place during burning is given by:



where the energy released is given off through (a) the measured heat of combustion associated with the energy released by molecular disassociation and not absorbed by the formation of new combustion products, and (b) the heat of explosion associated with the kinetic energy of the products of combustion. Previous tests using the laboratory bomb calorimeter have found that the heat of combustion from a mixture of S + KC1O₃ in the same proportions to that of the smoke is 385 calories per gram of mixture. Calculations outlined in appendix B indicate that the energy released in the form of heat of combustion from the reaction given in equation 1 assuming total combustion has a value of 377 calories per gram, in good agreement with the measured value. To date no known data are available for the heat of explosion of the reaction in equation 1. Since the energy released in burning of the smokes is in two forms, the heat energy was monitored by two different methods. The heat of combustion was absorbed by the sample holder and immediate surroundings and appeared primarily as an increase in the temperature of the steel sample holder (Sh) and the steel hang-down rod (hr) supporting the sample holder.

This energy H in calories is given by

$$H = 0.5555 MC \Delta T \quad (\text{Eq. 2})$$

Where: M = mass of the sample holder hand-down rod in grams

C = specific heat of steel in BTU/lb°F

ΔT = change in temperature in degrees Fahrenheit

The energy released in the heat of explosion was transmitted to the air in the closed vessel and appeared primarily as an increase in the static pressure of the air.

This energy in calories is given by:

$$H = 46.64 \frac{1}{\gamma-1} V \Delta P \quad (\text{Eq. 3})$$

Where:

γ = ratio of specific heat (c_p/c_v) of air in the tank

V = volume of the tank in cubic feet

ΔP = change in pressure in pounds per square inch

Analysis of the data on Violet Smoke IV shows that almost complete burning occurred within 30 seconds after ignition as seen in figure 2, with a maximum change in static pressure of 2.23 psig occurring inside the tank and a temperature change of the sample holder of 100°F.

Assuming uniform heating of the entire sample holder, the energy absorbed by the sample holder was 4.1 kilocalories. Since one end of the hang-down rod was thermally attached to the sample holder and the other end to the essentially constant temperature vessel wall it was assumed the average temperature rise of the rod was one half that of the maximum rise observed in the sample holder.

Using that assumption, the hang-down rod absorbed 2.5 kilocalories of heat. These calculations are outlined in appendix C. Therefore, the total energy released in the heat of combustion is 6.6 kilocalories, in good agreement with the heat release predicted in appendix B.

The heat energy present in the escaping gases was monitored, assuming efficient heat exchange to the air present in the vessel and assuming that the air acted as an ideal gas. It was further assumed that during this transfer no heat was lost to the wall of the tank. The measured change in pressure of 2.23 psig then corresponds to a change in internal energy of 9.8 kilocalories. The total energy released from 50 grams of the violet smoke is thus found to be 16.5 kilocalories. This compares to 149.7 kilocalories expected from complete oxidation of the sample (based on 2.994 Kcal/gm as the heat of combustion derived from tests in the laboratory Parr Bomb calorimeter). The decreasing exponential behavior of the temperature and pressure of the air after 25 seconds gives a measure of the heat transferred to the tank. From this variation and the fact that most of the burn occurs in about 15 seconds the loss is estimated to be about 1.5 to 2.0 kilocalories, or approximately 10 percent of the measured values, within the time of interest. With the possible variation in constituents shown in appendix D error limits of \pm 10 percent can be expected.

In the case of the green smoke IV, the burning time was approximately 60 to 70 seconds. Although the longer burning time allowed for more interaction between the two monitoring techniques and higher losses to the tank, the same methods and assumptions were used as were discussed for the violet smoke. The measured change in temperature of the sample holder of 140°F corresponds to an absorption of 5.8 kilocalories by the sample holder and 3.6 kilocalories by the hang-down rod. The measured change in pressure of the air of 1.50 psig corresponds to an increase of internal energy of 6.6 kilocalories. Therefore, the total measured energy release is 16.0 kilocalories. This value is lower than the measured value for the violet smoke due to the increased loss to the tank wall resulting from the longer burn time. This compares to 124.4 kilocalories expected from complete oxidation of the sample (based on 2.487 kcal/gm as the heat of combustion derived for green smoke when tested in the laboratory Parr bomb calorimeter). The higher measured value and the greater loss in comparison to the violet smoke is expected because of the nearly 10 percent more volatile ingredients (see appendix D). The larger value for the heat of combustion and corresponding smaller value of the heat of explosion is primarily due to the longer burn time which allowed for additional transfer of heat energy from the thermally excited escaping gas to the sample holder. Therefore, it appears that the degree to which the heat of explosion may be measured solely in terms of the air pressure is dependent on the length of burn time. It should be noted that the plateau observed in figure 3 in the pressure data around 30 seconds

is due to the reduction in burn rate caused by higher temperature and pressure. This effect has been observed in bomb calorimetry tests where the particular rate change varies with the smoke in question.

4.0 CONCLUSIONS

4.1 Impingement Studies. It is concluded that smoke compositions Violet IV and Green IV do not exhibit an explosive or burning reaction when impinged on a steel target set at 0 and 45 degrees to the direction of flight at velocities of 200, 250, and 300 feet per second.

The five components, sulfur, aluminum, potassium chlorate, benzathornc, and sodium bicarbonate shows no explosive or burning reaction when impinged on steel or aluminum targets set 45 degrees to the direction of flight at a velocity of 300 feet per second.

Sodium bicarbonate did exhibit an apparent electrostatic spark discharge when impinged on both steel and aluminum targets set at 45 degrees to the line of flight at velocities above 200 feet per second. There was no tendency for this spark to initiate a burning or explosive reaction.

Electrostatic investigations are inconclusive since charge measurements taken on the target during impingement showed no significant trends. The electrostatic charge measurement taken on target during impingement with aluminum dust was three orders of magnitude higher than the other materials used.

4.2 Friction Sensitivity Studies. It is believed that this apparatus can be used effectively to determine sensitivity of materials to friction forces. However, some additional modifications will be necessary before meaningful data can be accrued.

4.3 Calorimetric Studies. The total energy release in the two smoke compositions tested is approximately the same as would be expected since the same reaction is taking place. The measured energy released for a 50 gram sample is as follows:

- Violet IV B-143-5-1 = 16.5 kilocalories
- Green IV B-143-2-1 = 15.99 kilocalories

The energy release of 16.5 kilocalories released by the burning of 50 grams of violet smoke or equivalently 17 grams of its combustible mixture represents the closest value to the energy release for this type reaction, while remaining about 10 percent low.

5.0 RECOMMENDATIONS

5.1 Impingement Studies. It is recommended that impingement test apparatus used for investigating the characteristics of powdered or granular materials have a sample storage and injection system of sufficient capacity to achieve a uniform mixture and stable flow conditions simultaneously throughout the entire ejector tube. Stable flow conditions would provide a better measurement of air/material velocity. In addition, the continuous flow of material in the target area would more closely simulate the conditions expected in a factory processing system and provide a nucleus of material to which any induced electrostatic charge could transmit energy.

For a system such as that described above, velocity measurements should be made of the air flow driving the system and that parameter plus material flow rate used to define the processing limits for factory systems. The airflow and particle velocity capability should be increased by a factor of three in order to determine the fullest extent of the velocity safety margin.

A test program should be conducted using pelletized smoke compositions and mixtures of the various components of the smoke mix to test the effect of mass dependency and selective agglomeration on impingement characteristics.

An electrostatic charge measuring device should be designed that could be placed inside a pipe carrying a continuous flow of pyrotechnic material to determine the maximum charge accumulation under flow conditions in an ungrounded system. An electrostatic charge measuring device should be designed which could be placed at the exit end of the ejector tube to measure the dust cloud charge.

To counter the light tube effect on the optical sensors used to time the passage of dust over a known distance the sections of pipe on either side of the sensors and the mounting blocks should be made of metal or other opaque material.

To reduce the velocity measurement differences noted from test to test when no external factors were changed, it is recommended that a third set of optical sensors be added to the system providing two transit time measurements for each test. An investigation of existing laser equipment for the measurement of cloud velocity should also be undertaken.

It is believed that a pneumatic conveying system of a size commensurate with the needs of the pyrotechnic industries should be constructed for the purpose of conducting full scale flow and component tests. This system could be used to develop processing parameter limits, develop and test explosion or fire incident sensing devices. It would provide a test bed for determining electrostatic charges at critical points in the system and provide the capability of evaluating valves, mixers, separators and safety devices.

5.2 Friction Sensitivity Studies.

- Remount the sliding bar in a ball-bearing track to eliminate the sliding friction between the sliding bar and its support block.
- Modify the preload cell and preload screw bracket arrangement to provide an even preload over the entire top plate which encloses the sample material.
- Modify the pendulum release mechanism to reduce the friction in the solenoid retraction mechanism.

5.3 Calorimetric Studies. It is believed that the recommendations made below will improve the accuracy of the measured values, enable better correlation between measured and predicted values, and give a fuller picture of what reactions are taking place during the burning.

- Replace the steel hang-down rod with a less thermally conductive material capable of withstanding 600°F temperatures. This would decrease uncertainties concerning the hand-down rod.
- Place a second pressure transducer in the vessel symmetrically opposite the present instrument and utilize the average pressure of the two instruments in the calculations. This would minimize the effect of any pressure gradient which could occur during the burning processes.
- Place the gas sampling bottles directly on the vessel with as large a feed line as can be feasibly attached to the samplers to insure a more representative sample of the escaping gases.
- Weight the residue of the combustion processes to provide a better determination of the reactants consumed.
- Line the inside of the tank with a layer of insulation material to slow down the transfer of heat to the vessel walls.
- Insulate the outside of the tank to provide a more adiabatic test vessel.

REFERENCE DOCUMENTS LIST

1. Project Plan for Hazards Evaluation Processing Indices, PL-4D11.
2. Design Specification for Impingement Reaction Apparatus, 1E101A33002.
3. Acceptance Test Procedure for Impingement Reaction Apparatus, TWR No. EA-4D11.
4. Program Memorandum, Acceptance Test Impingement Reaction Apparatus, 74-13-001.
5. Test Procedure for Impingement Reaction Apparatus, GE-HERE-400.
6. Program Memorandum, Impingement Reaction Apparatus Pyrotechnic Classification Tests, 74-15-001.
7. Fabrication Drawing Package Impingement Reaction Apparatus, 15 sheets, Dwg. No. EA-4D11.
8. Specification for Friction Sensitivity Apparatus, 1E101A33001.
9. Acceptance Test Procedure for Pendulum/Friction Sensitivity Device, 22-EA-4D11, Sub. 002.
10. Test Procedure for Friction Sensitivity Device, GE-HERE-TP-407.
11. Program Memorandum, Friction Sensitivity of Pyrotechnic Smoke, 74-25-001, Appendix - Hazardous Material Laboratory Report
12. Fabrication Drawing Package, Friction Sensitivity Apparatus, 1E501D33011.
13. Test Procedure Instruction for Use of the 37 Cubic Foot Parr Bomb Vessel, GE-HERE-TP-408, Appendix - Hot Wire Firing Device, and Blasting Device Circuits, Tank Instrumentation.
14. Program Memorandum, Modified Parr Bomb Tests, 74-28-001, Appendix - Hazardous Material Laboratory Report, Charge Holder for Hot Wire Device

APPENDIX A - FIGURES

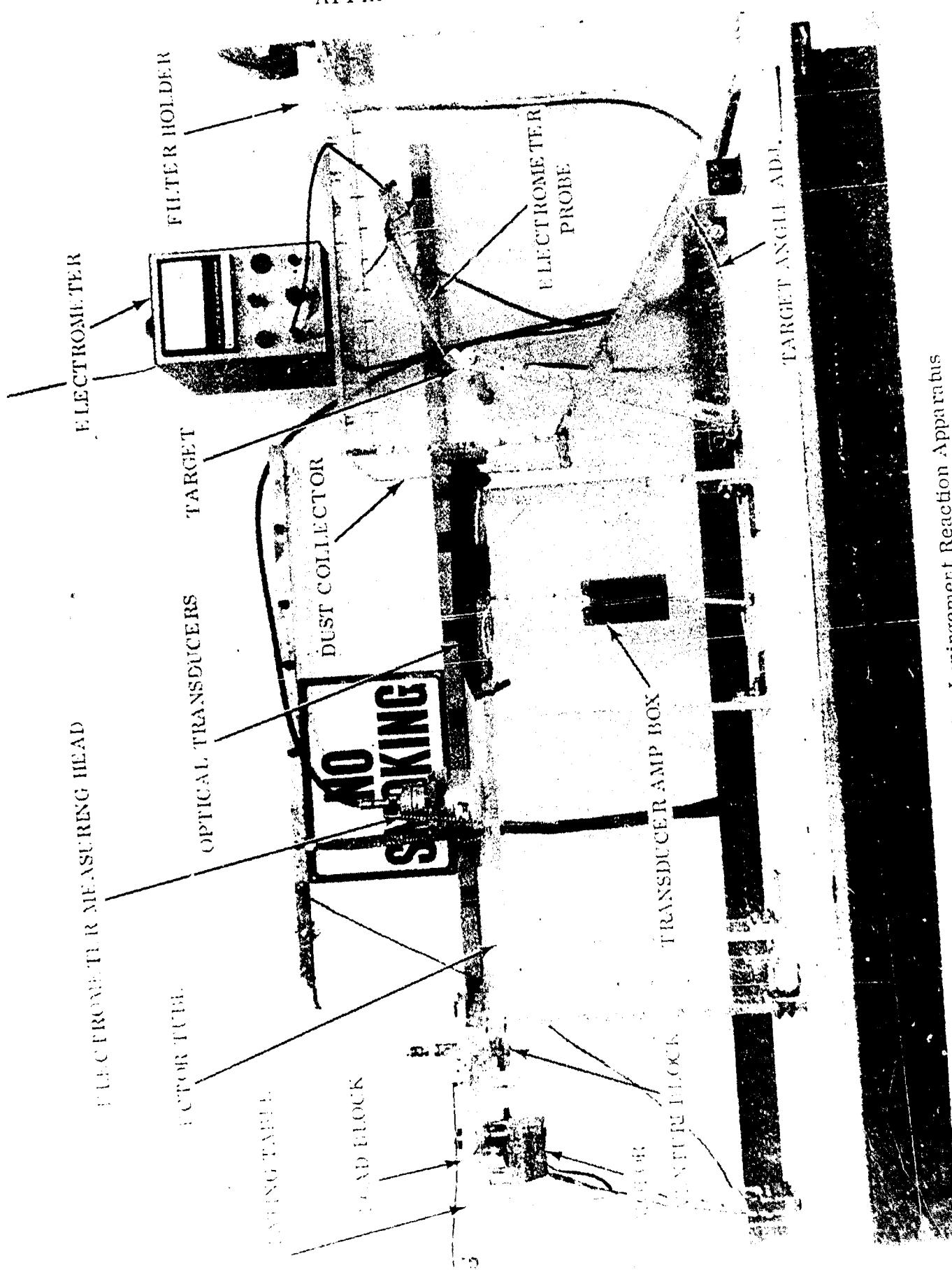


Figure A-1. Impingement Reaction Apparatus

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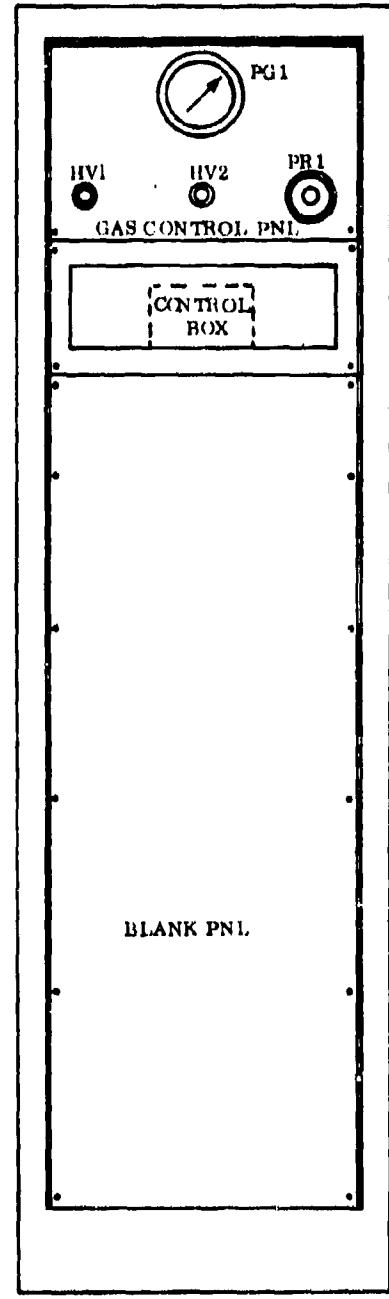
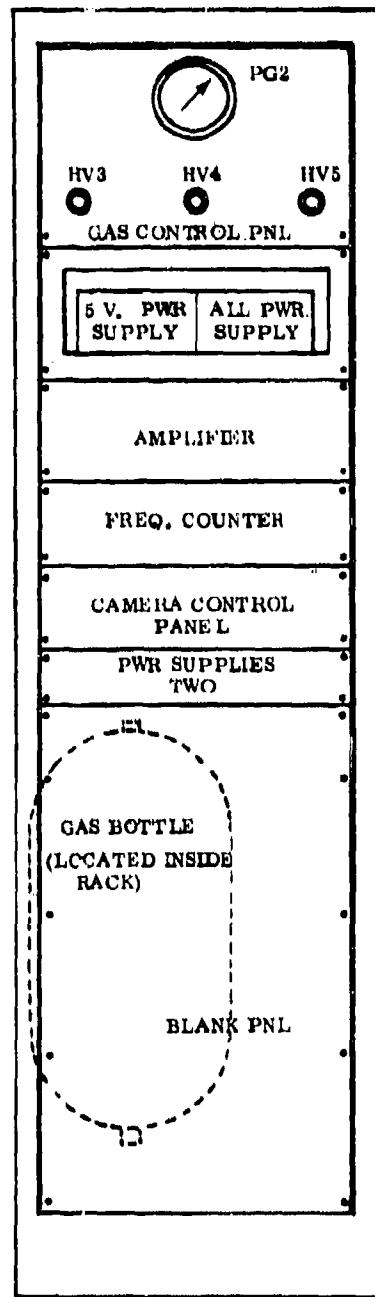
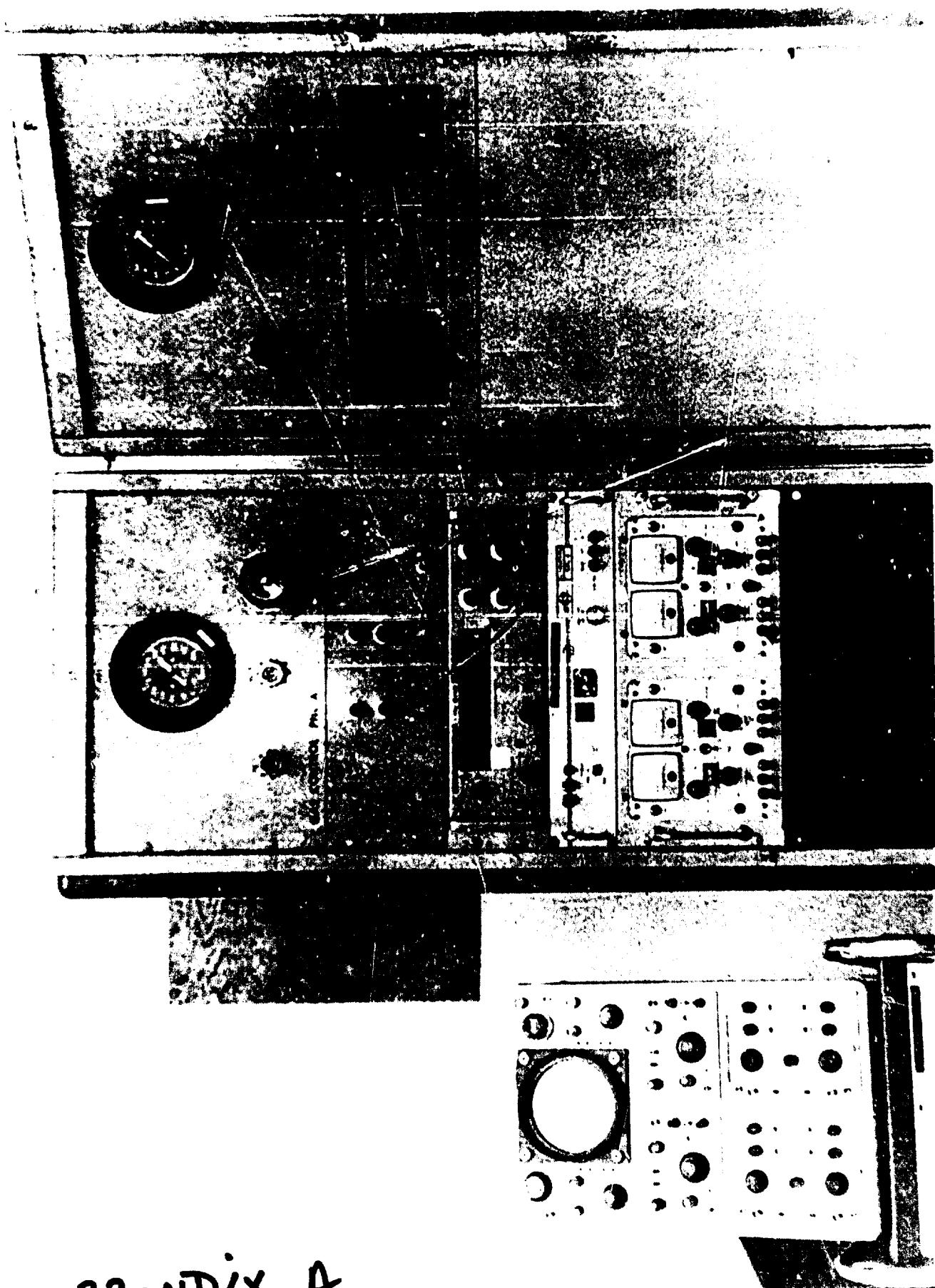


Figure A-2. Instrumentation and Gas Control Cabinets for Impingement Apparatus

Figure A-3. Instrumentation and Gas Control Cabinets



APPENDIX A

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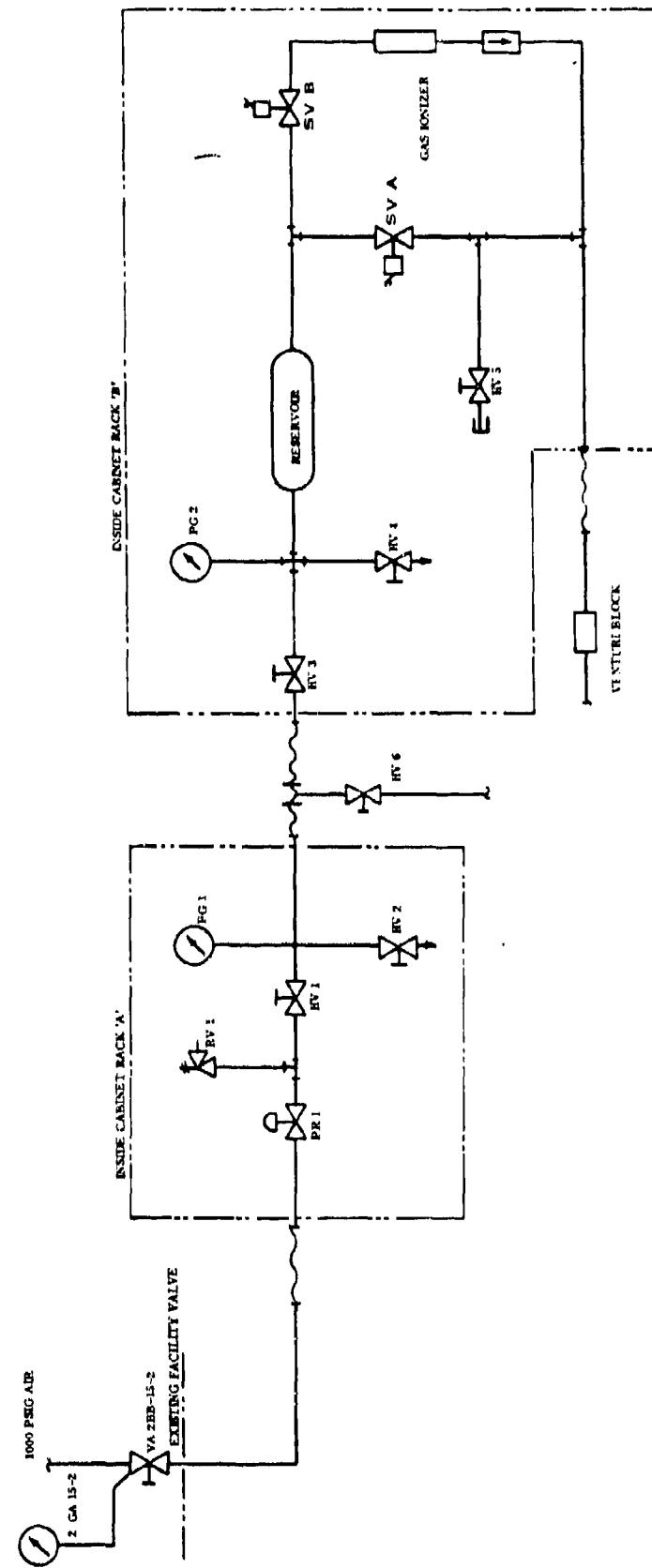


Figure A-4. Gas Flow Piping Schematic for Impingement Apparatus

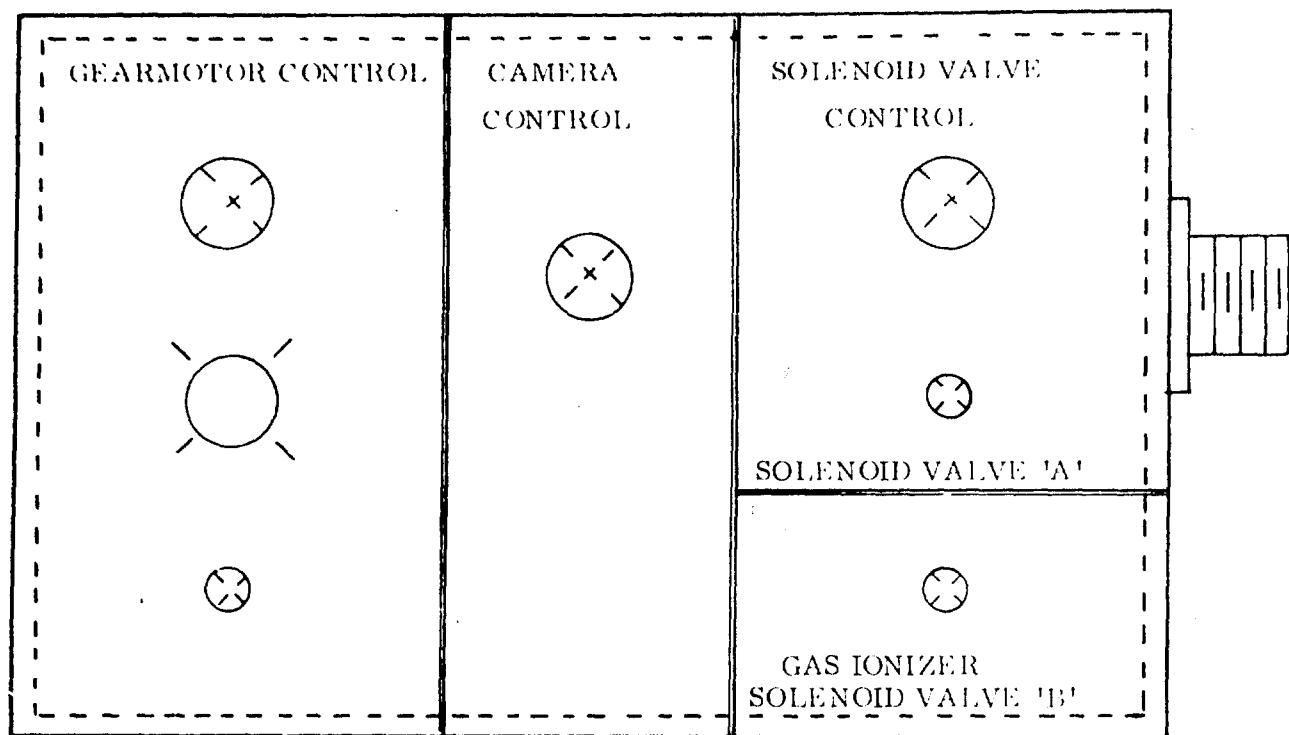


Figure A-5. Portable Gas Control Box For Impingement Apparatus

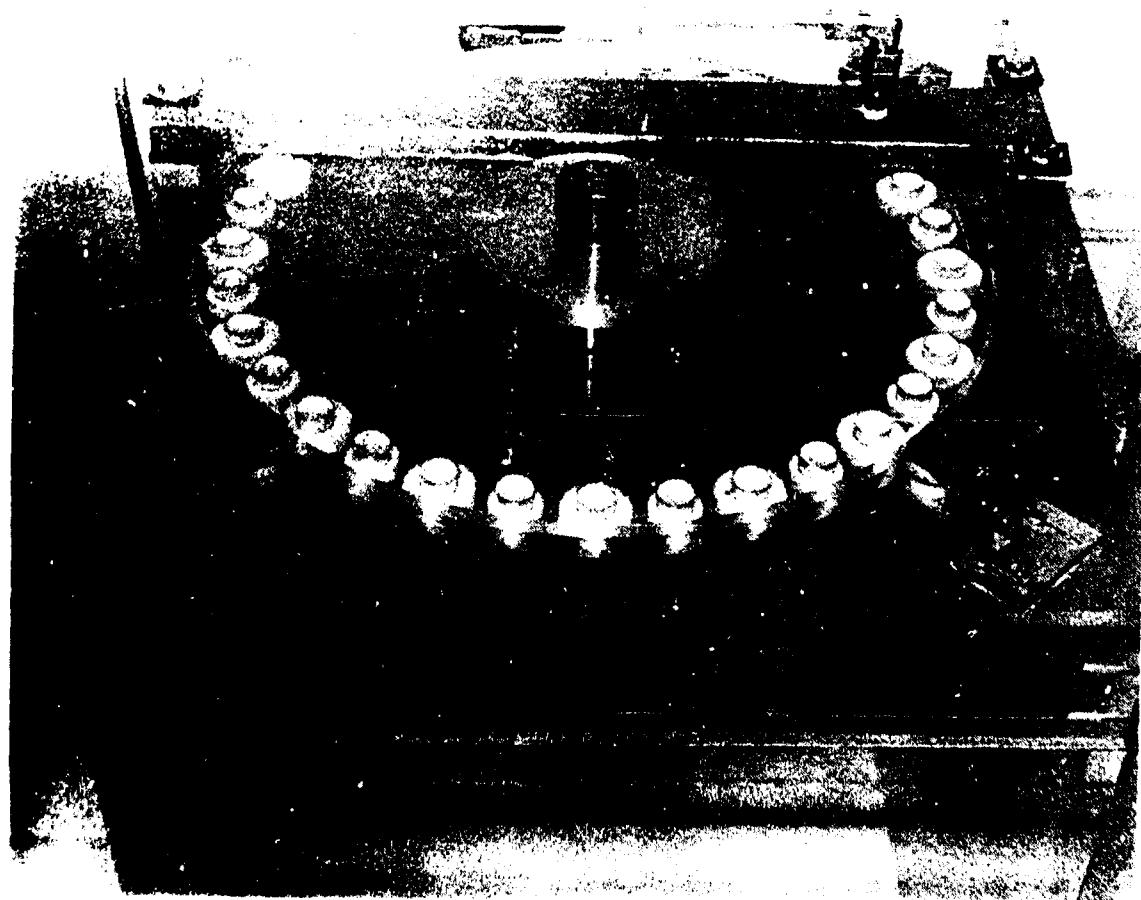


Figure A-6. Impingement Reaction Apparatus Showing the Rotating Sample Storage Table

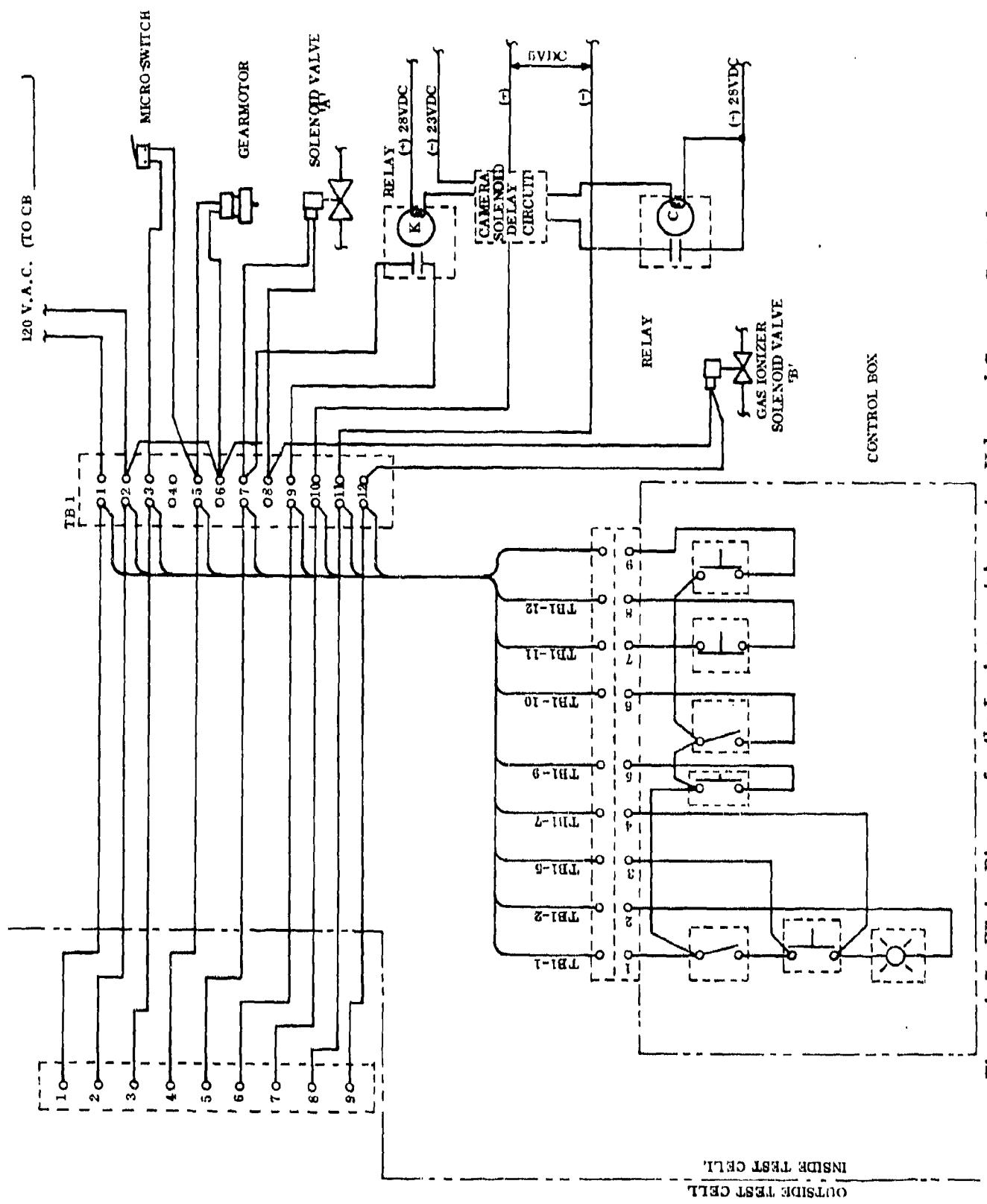


Figure A-7. Wiring Diagram for the Impingement Apparatus Valve and Camera Control Box

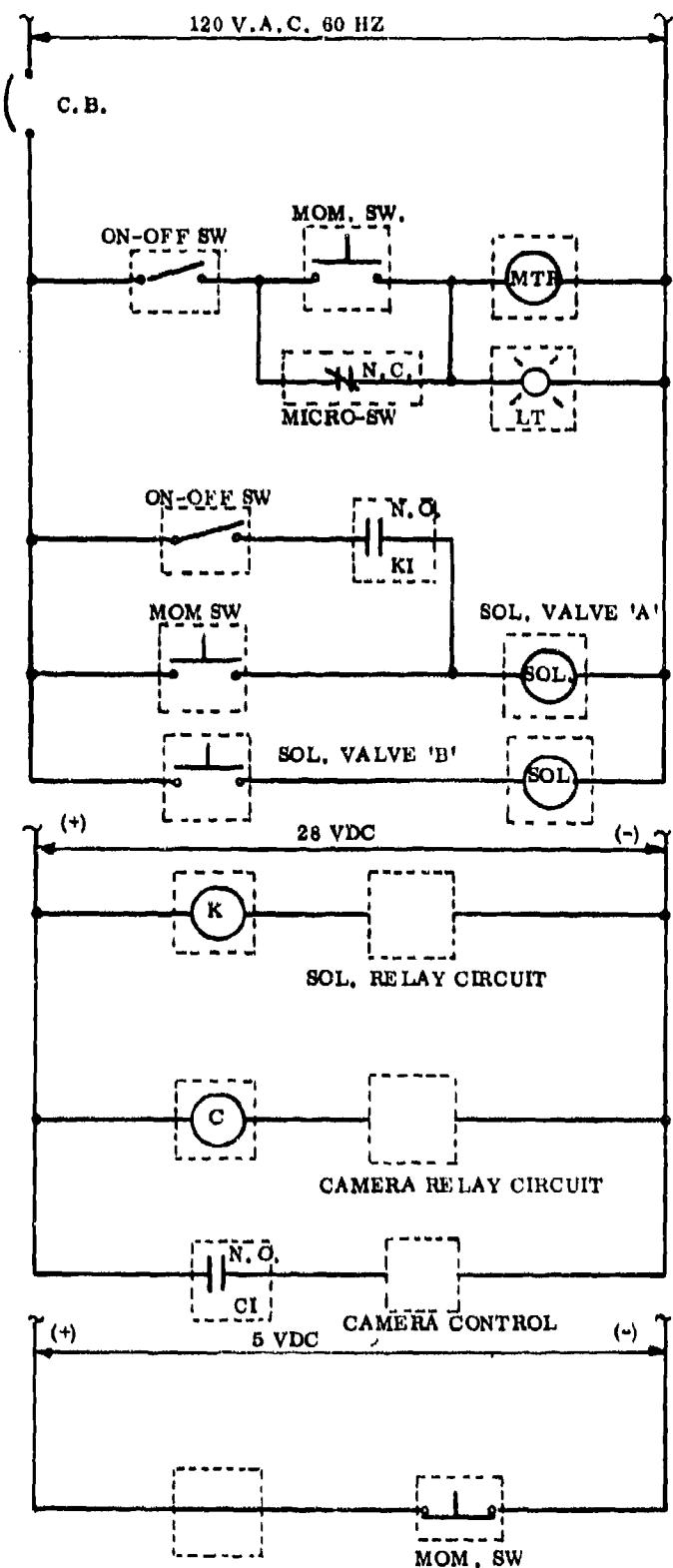


Figure A-8. Camera and Solenoid Delay Circuit Wiring Schematic

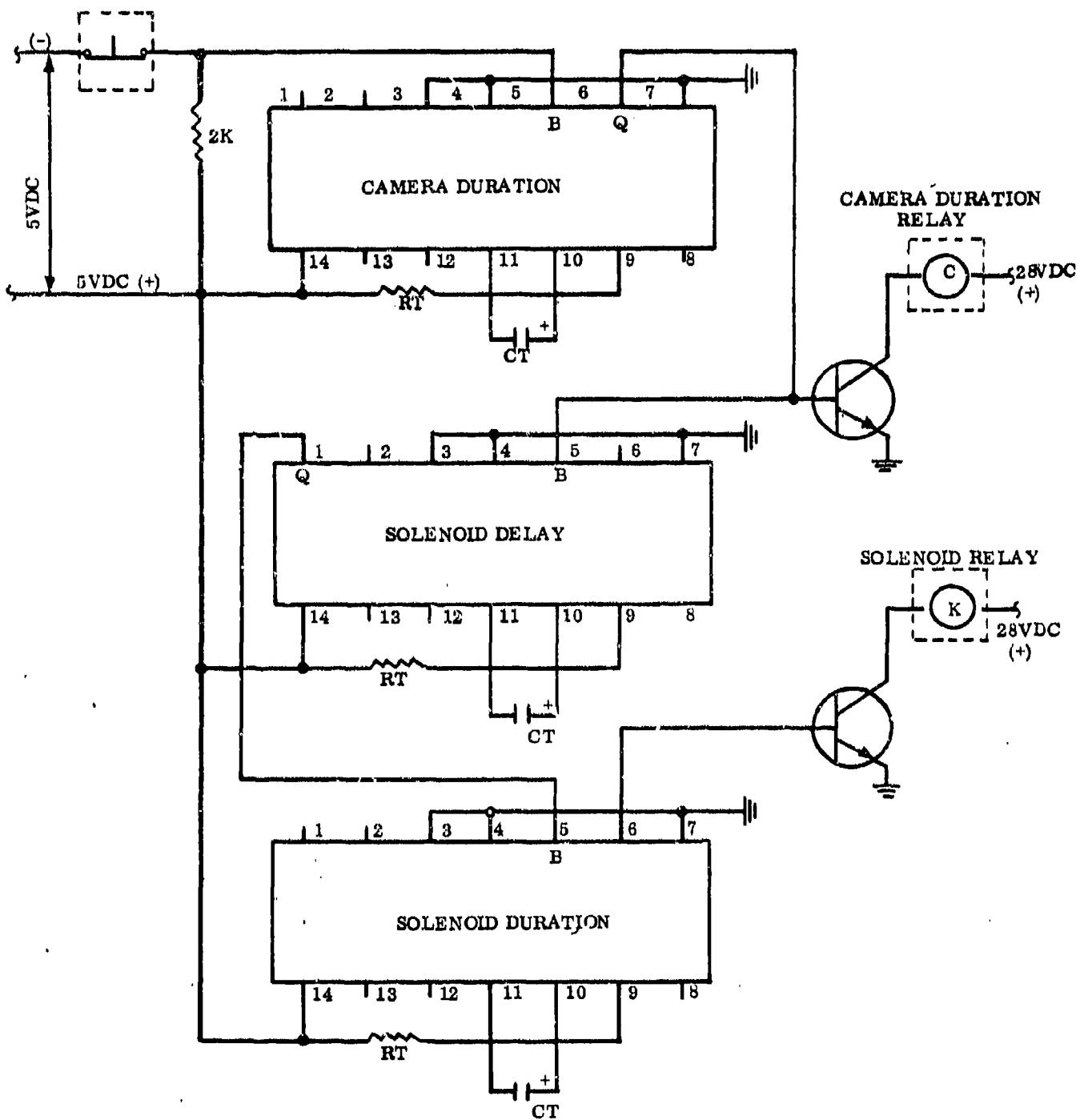


Figure A-9. Camera & Solenoid Delay Circuit Wiring Diagram for the Impingement Apparatus

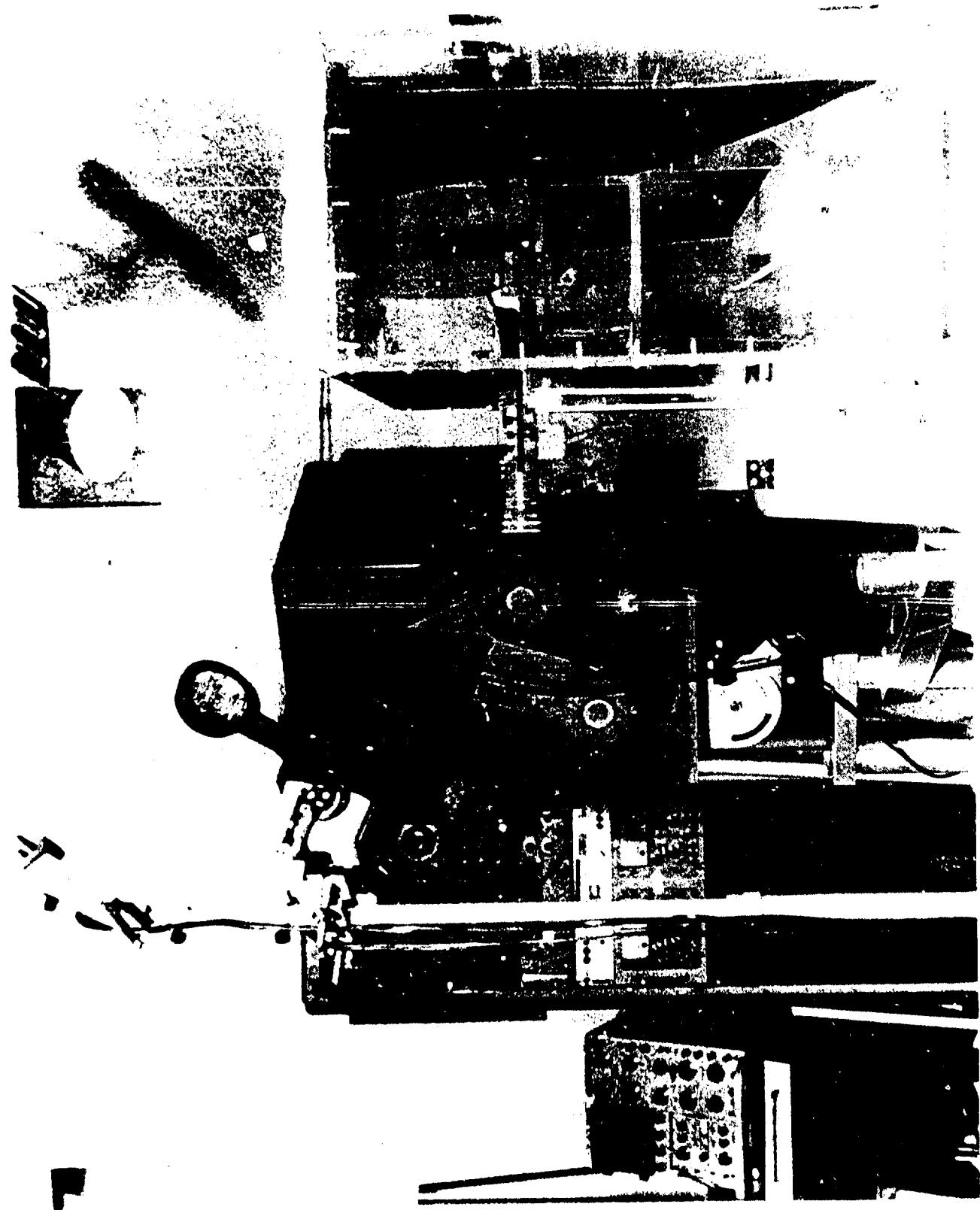


Figure A-10. Fastex Camera Setup for High Speed Motion Pictures

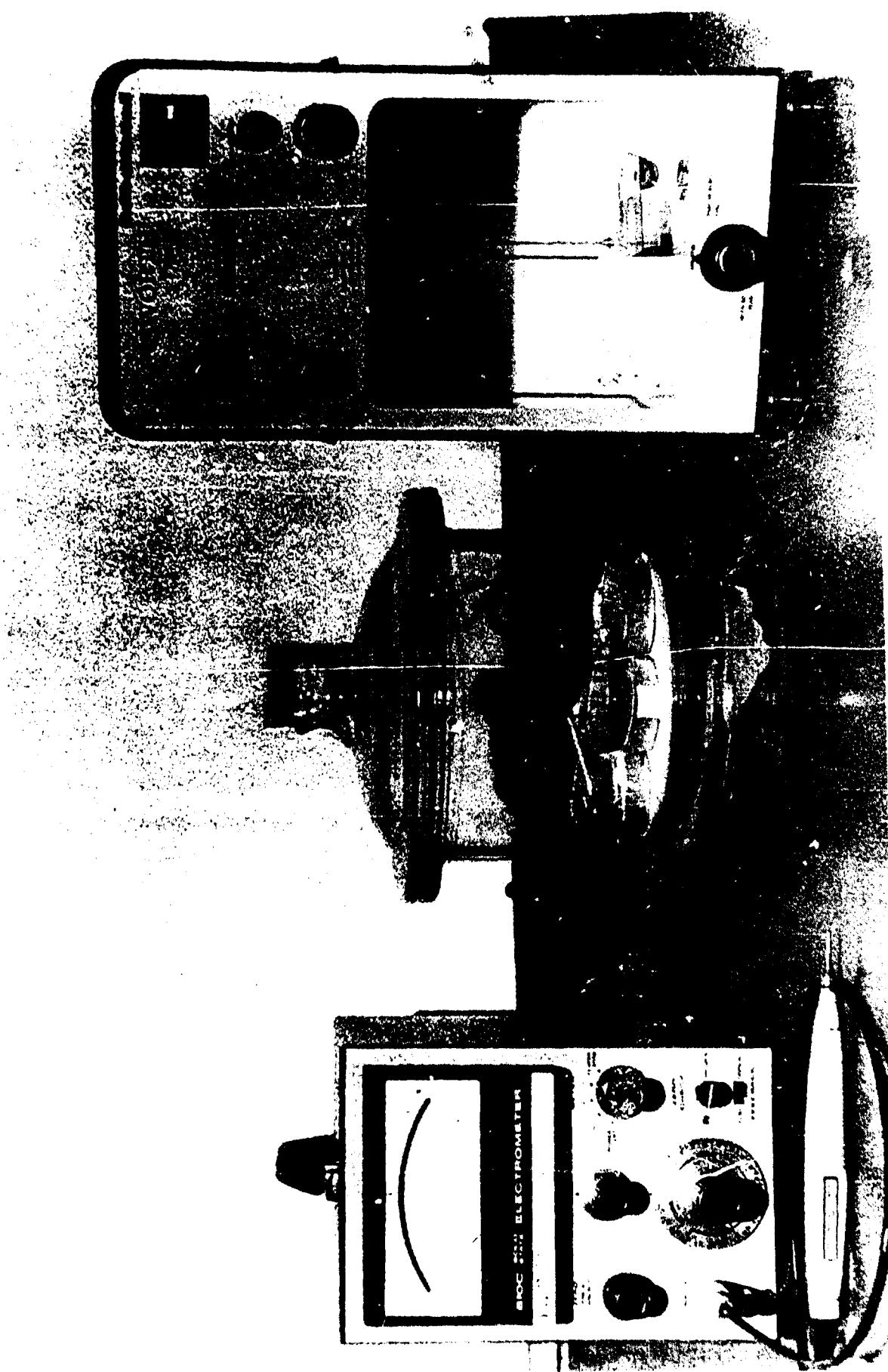
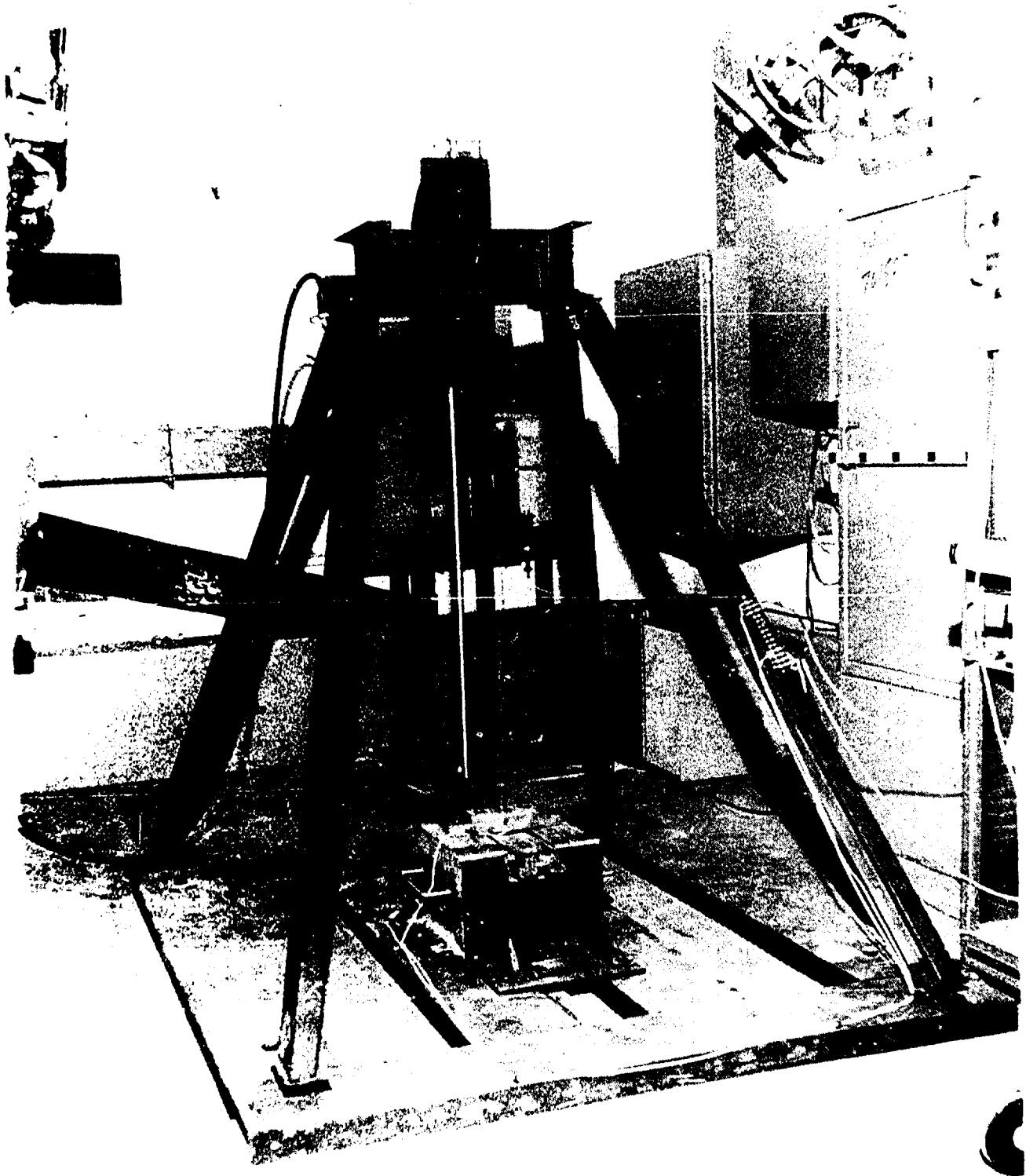


Figure A-11. Sample Preparation Bench Showing the Kiethly Electrometer, Desiccator and Balance for Sample Storage & Weighing



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Figure A-12. Friction Sensitivity Apparatus End View

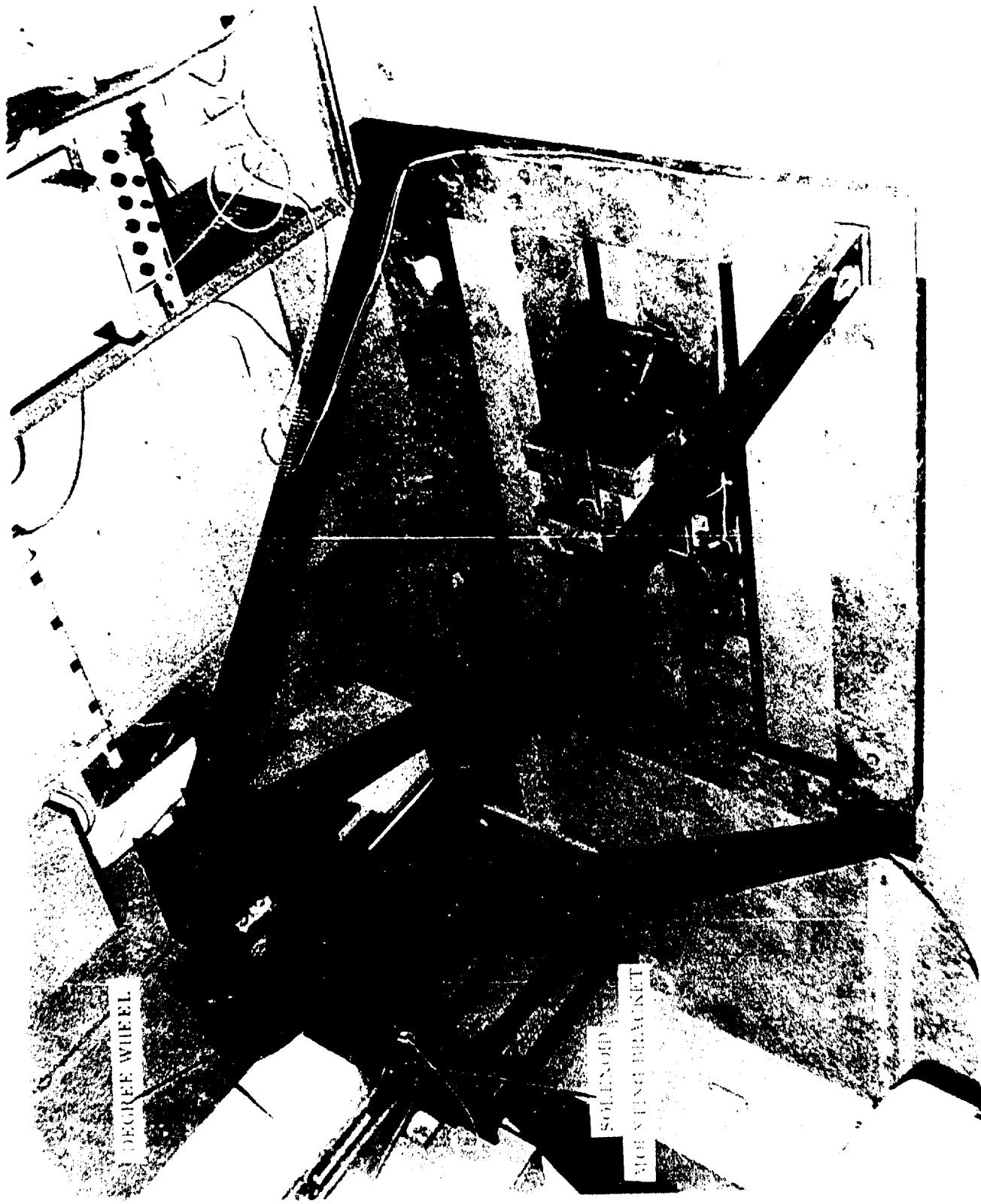


Figure A-13. Friction Sensitivity Apparatus Top View

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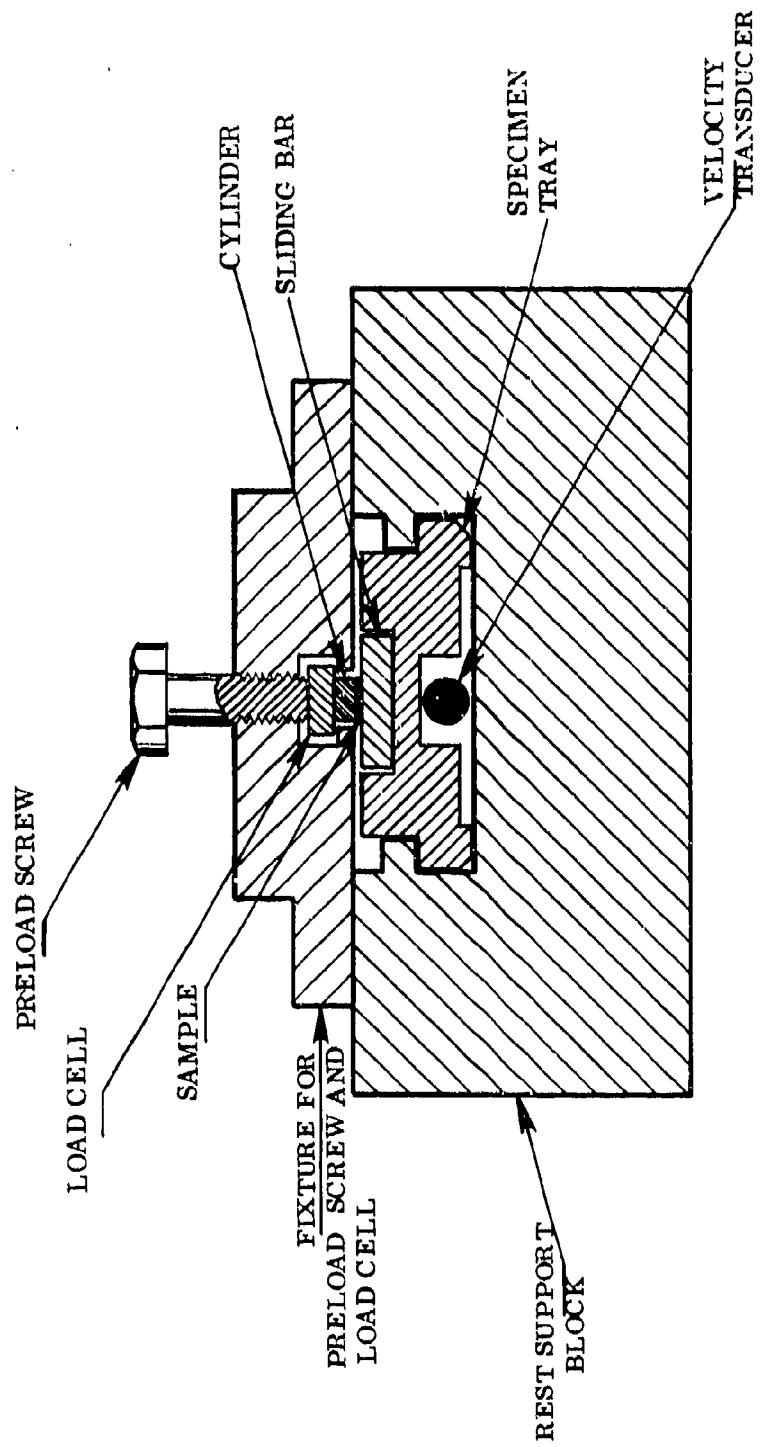


Figure A-14. Schematic of Material Holder

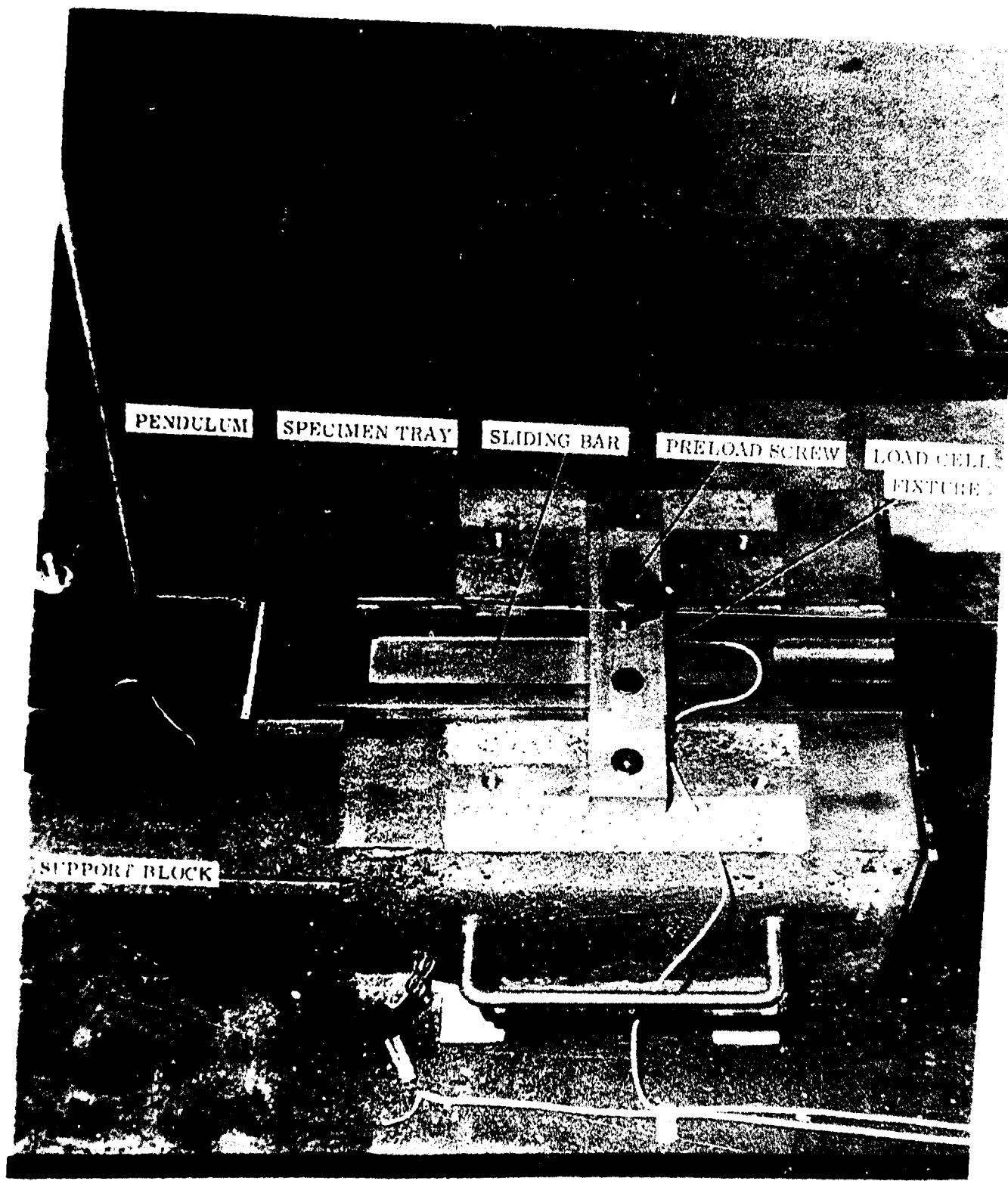


Figure A-15. Side View of Friction Sensitivity Apparatus Material Holder

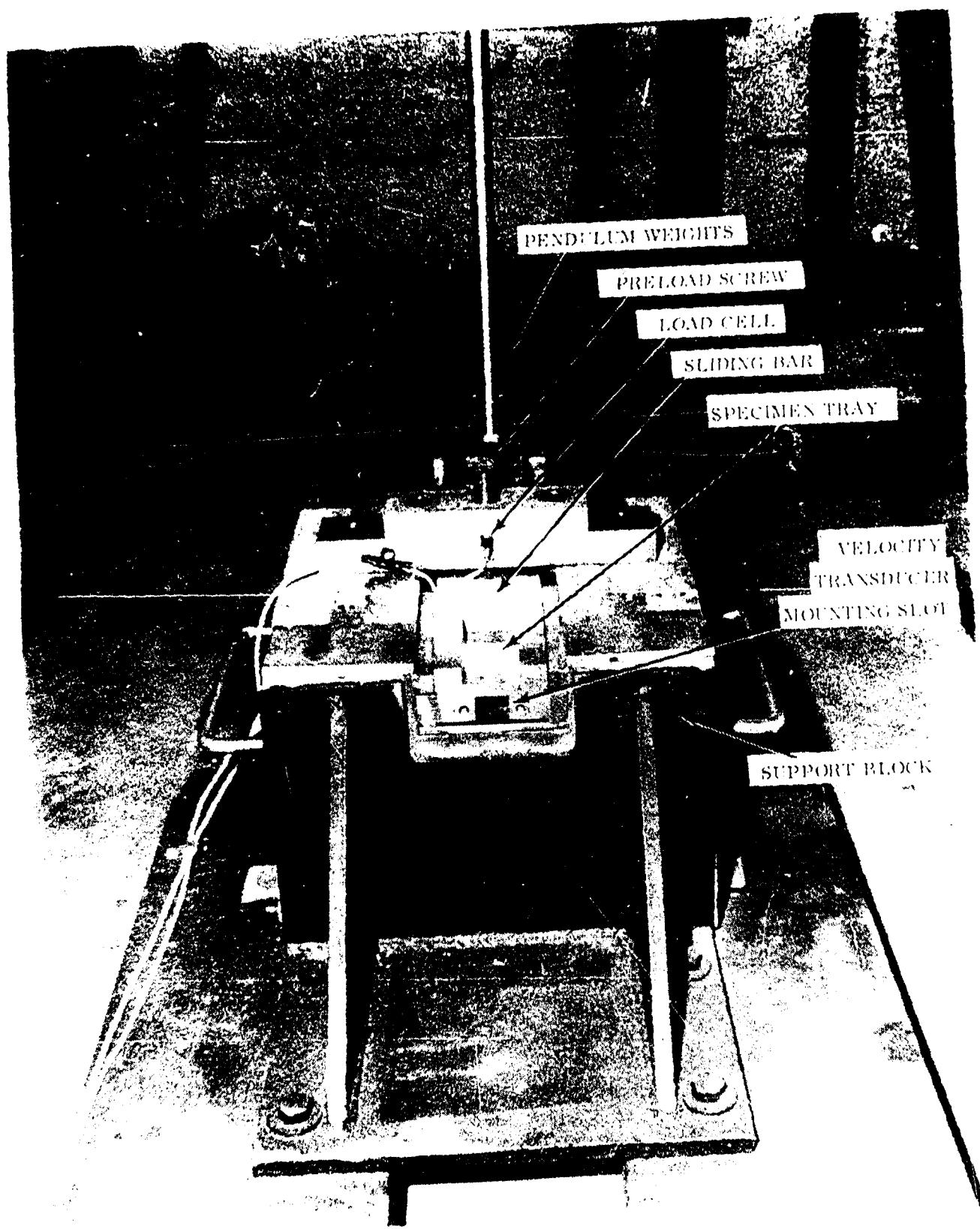


Figure A-16. End View of Friction Sensitivity Apparatus Material Holder

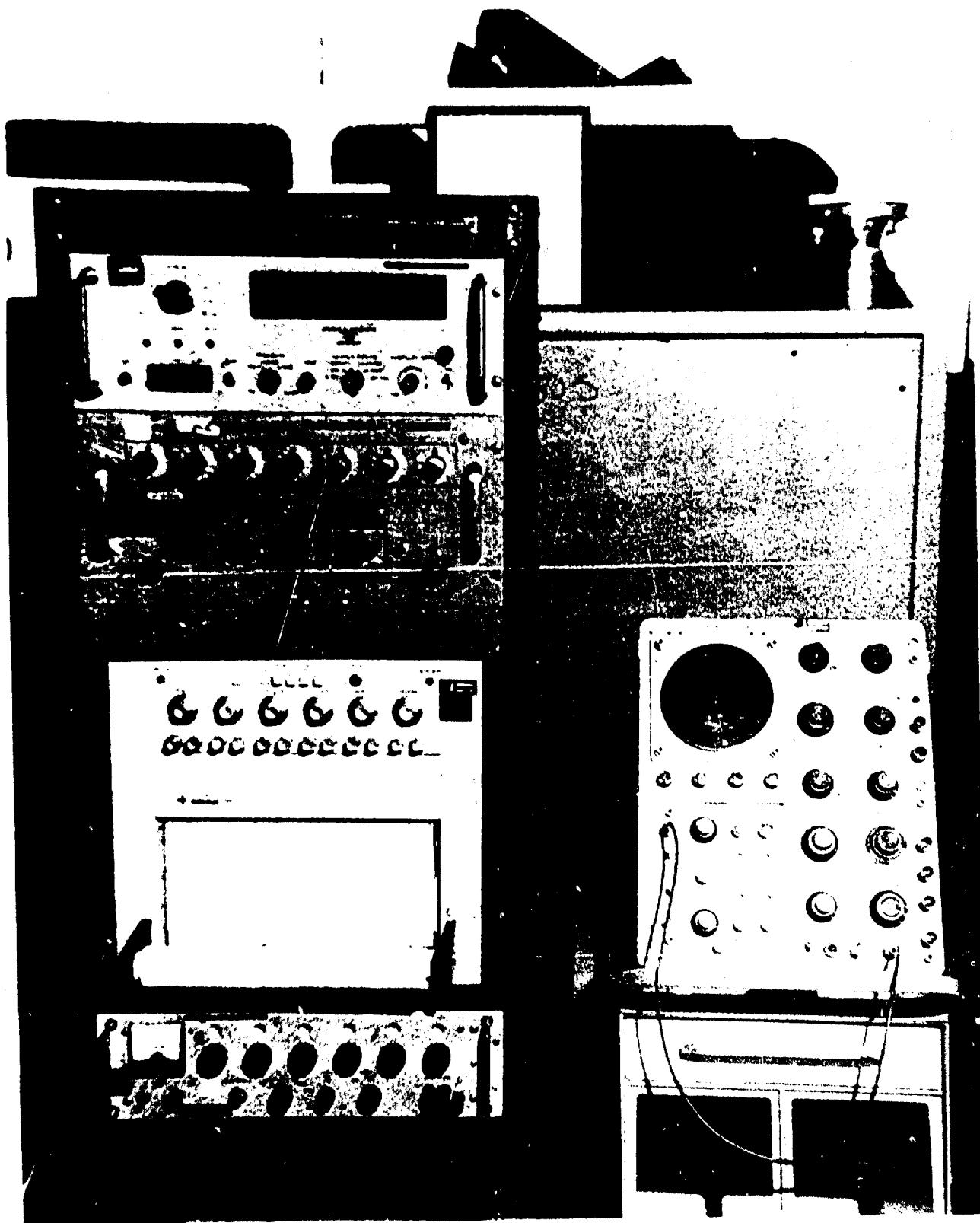


Figure A-17. Friction Sensitivity Apparatus Instrumentation Set Up
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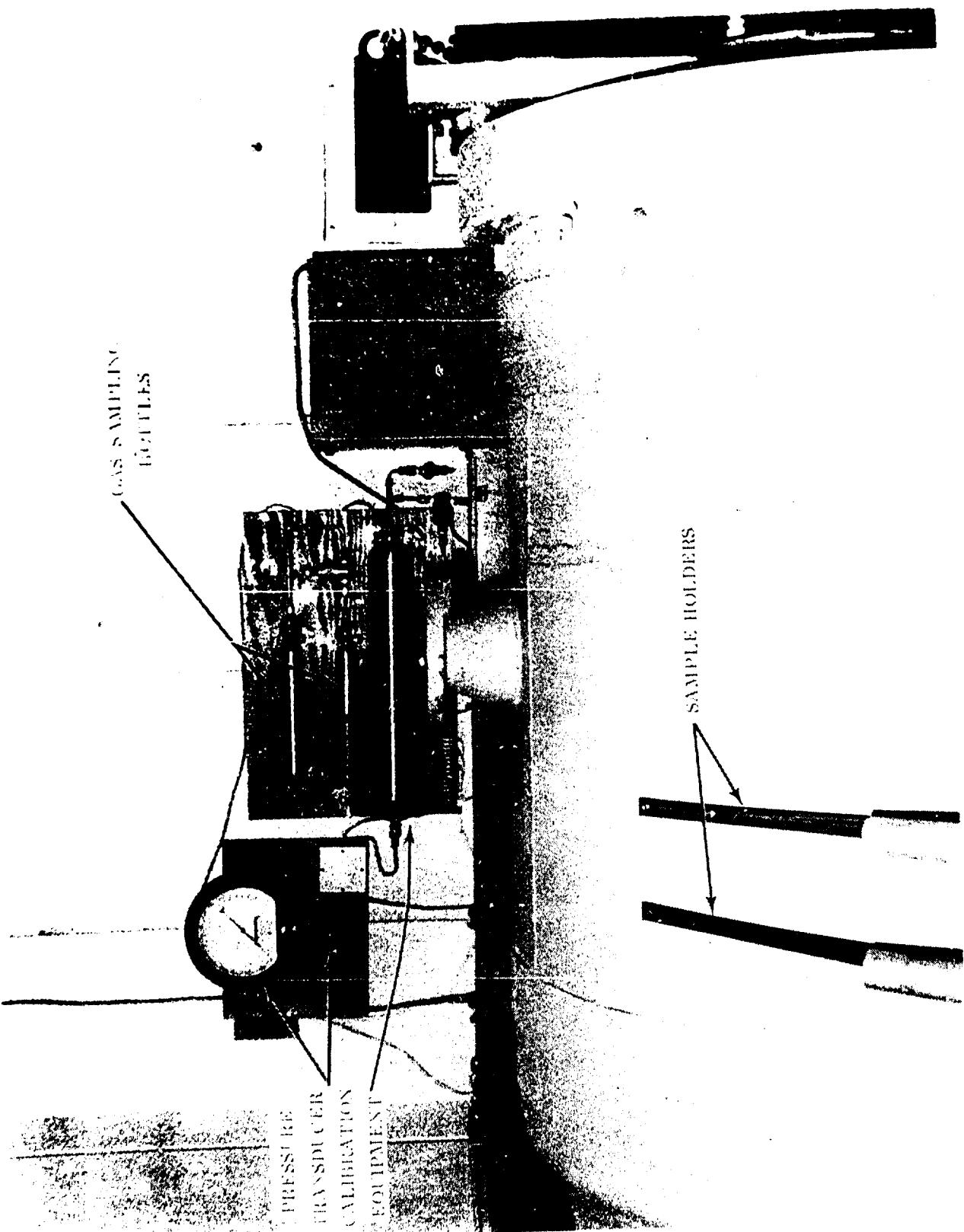


Figure A-18. 37.66 Cubic Foot Test Vessel

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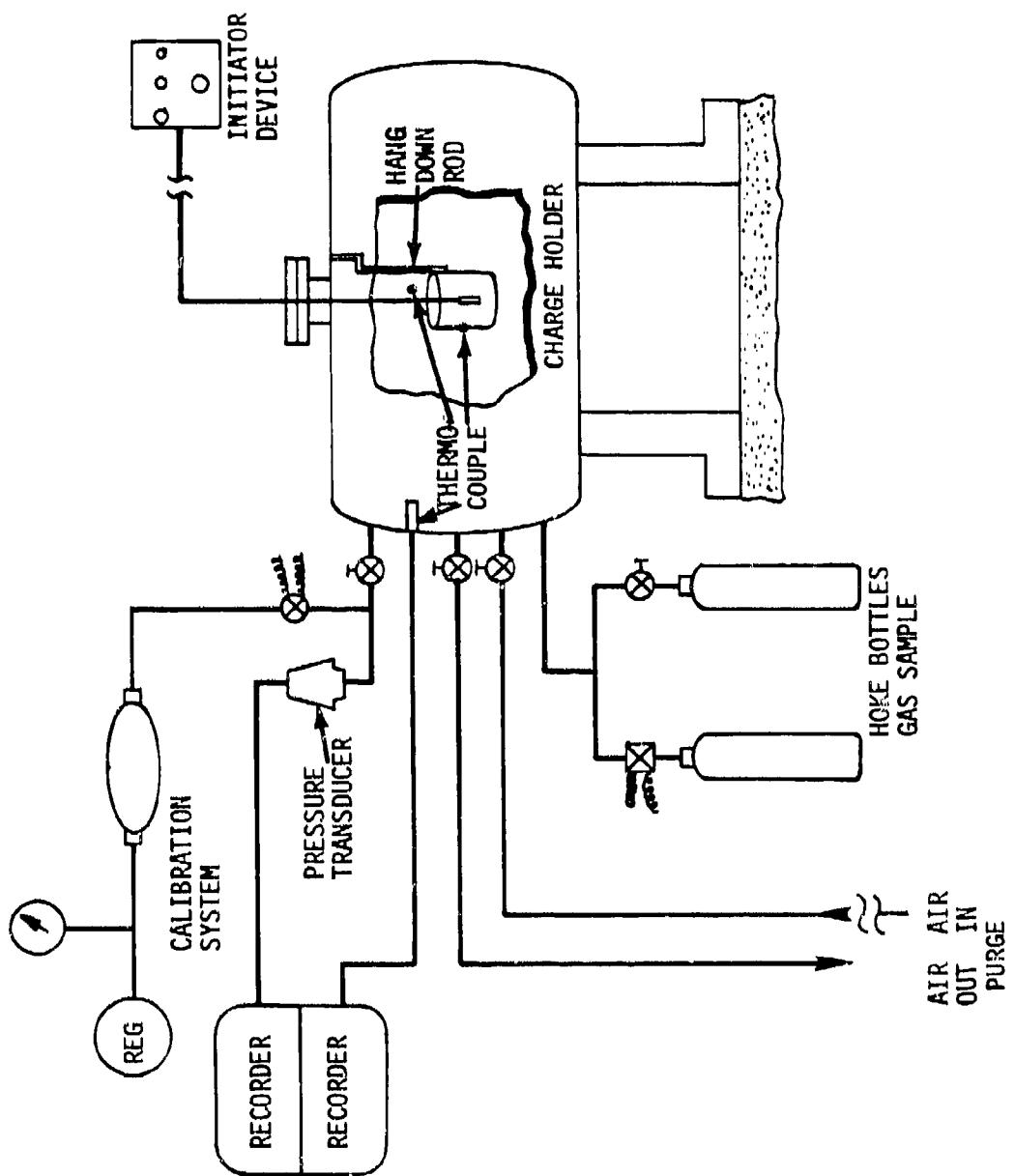


Figure A-19. Test Tank for Modified Parr Bomb Test Showing Instrumentation Set Up

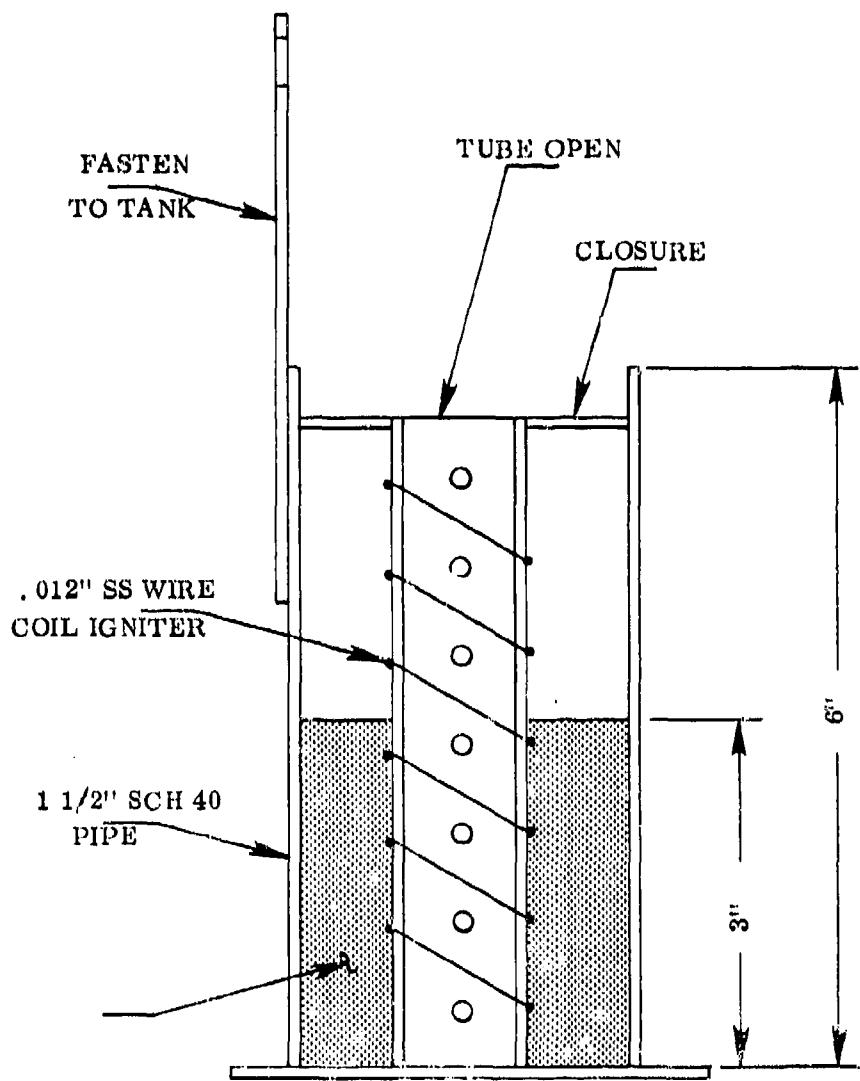


Figure A-20. Sample Holder Used to Burn Material in Closed Vessel Test

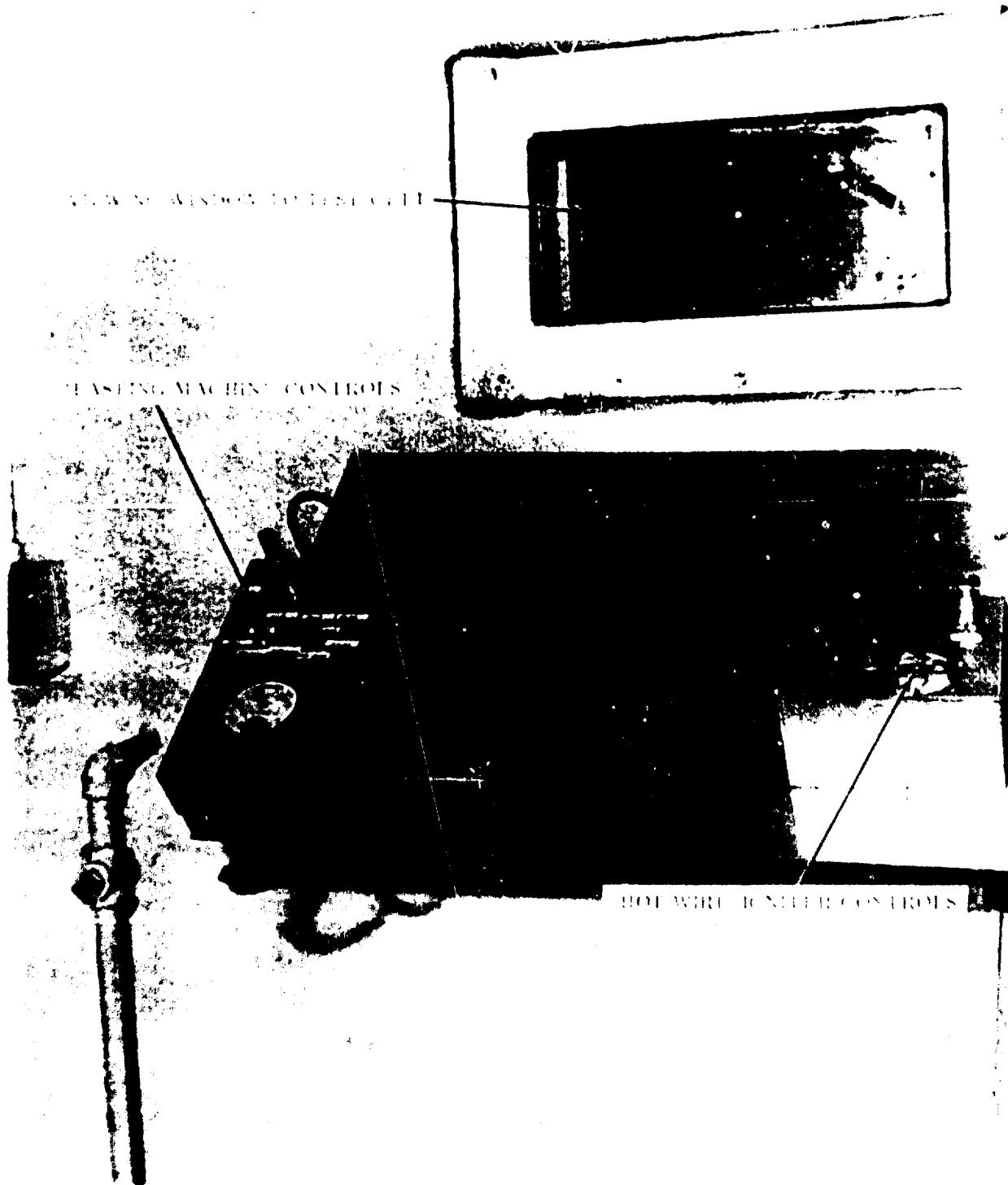


Fig. 11. Control station showing simple button control boxes.

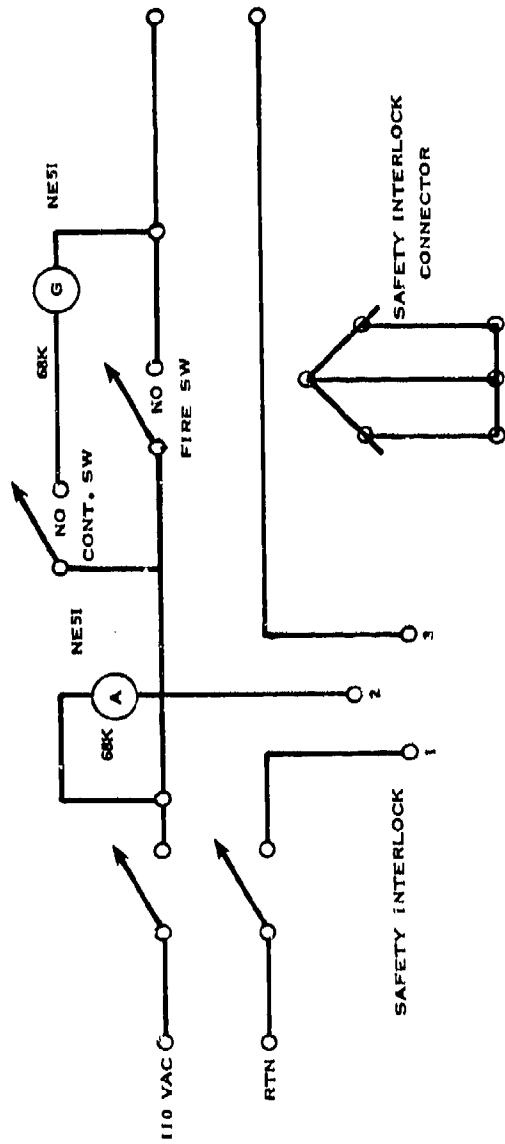
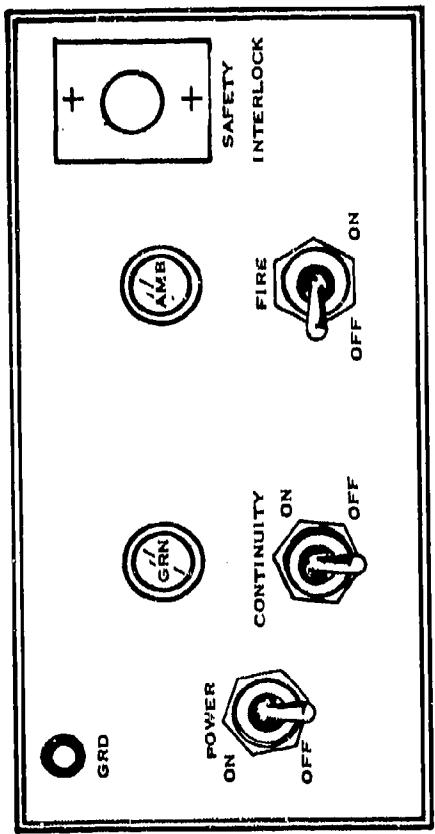


Figure A-22. Hot Wire Firing Device Schematic

APPENDIX B

CALCULATION OF HEAT OF COMBUSTION

Concerning the heat of combustion released in the chemical reaction described by



the following heats of formations obtained from the CRC Handbook of Chemistry and Physics were used.

| <u>Material</u> | <u>Heat of Formation</u> | <u>Gram Molecular Weight</u> |
|-------------------|--------------------------|------------------------------|
| S | 0.0 Kcal/mole. | 32.1 gm |
| KC1O ₃ | 93.5 Kcal/mole. | 122.5 gm |
| SO ₂ | 70.9 Kcal/mole. | |
| KC1 | 51.6 Kcal/mole. | |

For the case of 50 grams of violet smoke where 9 percent of the mixture is sulfur and 25 percent is potassium chlorate, the energy released in the reaction utilizing all of the sulfur and 0.093 moles of the .102 mole present of KC1O₃ results in an energy release of 6.0 K calories as shown below.

| | |
|--|------------------|
| Formation of .140 mole of SO ₂ releases | 9.92 Kcal |
| Formation of .093 mole of KC1 releases | 4.80 Kcal |
| Disassociation of .093 mole of KC1O ₃ absorbs | <u>8.72 Kcal</u> |
| | 6.00 Kcal |

This represents complete burning of 4.5 grams of sulfur and 11.4 of the 12.5 grams of potassium chlorate. Considering only the burning mixture, the heat of combustion represents 377 calories/gram in excellent agreement with the measured value of 385 calories/gram.

APPENDIX C

CALCULATION OF TOTAL HEAT RELEASE

A. Violet Smoke

Test #30-74-02

$$\begin{aligned}
 H(\text{explosion}) &= 46.64 \frac{\text{Kcalories}}{\text{psi} \cdot \text{ft}^3} \cdot \frac{1}{1.4-1} \cdot 2.23 \text{ psi} \times 37.66 \text{ ft}^3 \\
 &= 9.79 \text{ Kcalories} \\
 H(\text{combustion}) &= H_{\text{sh}} + H_{\text{hr}} \\
 H_{\text{sh}} &= 0.555 \frac{{}^{\circ}\text{C}/{}^{\circ}\text{F}}{\text{gm}} \times 617 \text{ gram} \times .12 \text{ cal/gm}^{\circ}\text{C} \times 100^{\circ}\text{F} \\
 &= 4.110 \text{ Kcalories} \\
 H_{\text{hr}} &= 0.555 \frac{{}^{\circ}\text{C}/{}^{\circ}\text{F}}{\text{gm}} \times 770 \text{ gram} \times .12 \text{ cal/gm}^{\circ}\text{C} \times 1/2 \times 140^{\circ}\text{F} \\
 &= 2.560 \text{ Kcalories} \\
 H(\text{combustion}) &= 6.670 \text{ Kcalories} \\
 \text{NOTE: } 17 \text{ grams of S + KC1O}_3 &\text{ with a heat of combustion of 385 cal/gram} \\
 &\text{releases 6.54 Kcalories} \\
 \text{Total Heat Released} &= (9.79 + 6.67) \text{ Kcalories} \\
 &= 16.46 \text{ Kcalories}
 \end{aligned}$$

B. Green Smoke

Test #30-74-004

$$\begin{aligned}
 H(\text{explosive}) &= 46.64 \frac{\text{Kcalories}}{\text{psi} \times \text{in}^3} \cdot \frac{1}{1 - 1} \cdot 1.50 \text{ psi} \times 37.66 \text{ ft}^3 \\
 &= 6.590 \text{ Kcalories} \\
 H(\text{combustion}) &= H_{\text{sh}} + H_{\text{hr}} \\
 H_{\text{sh}} &= 0.555 \frac{{}^{\circ}\text{C}/{}^{\circ}\text{F}}{\text{gm}} \cdot 617 \text{ gram} \cdot 0.12 \frac{\text{calories}}{\text{gm}^{\circ}\text{C}} \cdot 141^{\circ}\text{F} \\
 &= 5.800 \text{ Kcalories} \\
 H_{\text{hr}} &= 0.555 \frac{{}^{\circ}\text{C}/{}^{\circ}\text{F}}{\text{gm}} \cdot 770 \text{ gram} \cdot 0.12 \frac{\text{calories}}{\text{gm}^{\circ}\text{C}} \cdot 70^{\circ}\text{F} \\
 &= 3.600 \text{ Kcalories} \\
 H(\text{combustion}) &= 9.400 \text{ Kcalories}
 \end{aligned}$$

APPENDIX C (CONTINUED)

NOTE: 18.5 grams of S + KC₁₀₃ with a heat of combustion of 385 cal/gram releases
7.12 Kcalories

$$\begin{aligned}\text{Total Heat Release} &= (6.59 + 9.40) \text{ Kcalories} \\ &= 15.99 \text{ Kcalories}\end{aligned}$$

APPENDIX D

ADDITIONAL DATA AND PHYSICAL PARAMETERS USED IN THE CALCULATIONS

γ (air) = 1.40

c (steel) = 0.12 Kcalories/gram $^{\circ}$ C

Dimensions of Steel Pipe (Sample Holder):

| | | | | | |
|-------------|---|-------------|---------|---|-------------------------|
| O.D. | = | 1.90 inches | Volume | = | 78.6 cm ³ |
| I.D. | = | 1.61 inches | Density | = | 7.84 gm/cm ³ |
| Length | = | 6.00 inches | Mass | = | 617 grams |
| Weight/Foot | = | 2.17 lb | | | |

Dimensions of Hangdown Rod:

1/4" x 1" x 24"

Density = 7.84 gm/cm³

Mass = 770 grams

Smoke Specifications (% by Weight):

| <u>Material</u> | <u>Violet IV</u> | <u>Green IV</u> |
|--------------------|------------------|-----------------|
| Sulphur | 9.0 \pm 1.0 | 10.4 \pm 1.0 |
| Potassium Chlorate | 25.0 \pm 2.0 | 27.0 \pm 2.0 |
| Sodium Bicarbonate | 24.0 \pm 2.0 | 22.6 \pm 2.0 |
| Violet Dye | 42.0 \pm 1.0 | -- |
| Solvent Green Dye | -- | 28.0 \pm 1.0 |
| Yellow Dye | -- | 4.0 \pm 0.5 |
| Benzanthrone | -- | 8.0 \pm 0.5 |

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